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1933-1934

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REPORTS ON THE PROGRESS OF NAPHTHOLOGY, 1930-1931.

These reports are available bound in one volume, and can be obtained from the offices of the Institution at 10s. 6d. per copy.

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Advertisements. Advertisements are inserted in the Journal, and information as to terms, etc., can be obtained from the Advertising Manager, at the offices of the Institution, Aldine House, Bedford Street, W.C. 2. (Telephone No. Temple Bar 1842.)

Prepaid small advertisements, such as situations vacant and wanted, patents for sale and miscellaneous, are accepted at a charge of 1s. per line of seven words (minimum 4s.). Box number 1s. extra. These should be sent to the Associate Editor not later than the 12th of the month in which it is desired they should appear.

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PERSONAL NOTES OF MEMBERS AND SPECIAL NOTICES.

It is suggested that members send information regarding their movements to the Secretary, for insertion under this heading.

Mr. C. T. Barber has returned from Burma.

Mr. E. R. Cartwright is home from Singapore.

Mr. G. Dickinson has returned from Sarawak.

Mr. C. H. S. Edmonds is in France.

Mr. G. C. F. Greant has returned from Belgium.

Mr. C. S. Lee has left for Trinidad.

Mr. K. B. Ross is in Persia.

Mr. G. F. Wallis is home from Mexico.

The Secretary would be glad to learn of the whereabouts of the following members:—E. E. G. Agius, G. Bourkoff, A. F. Brown, A. E. C. Corbet, A. R. Crawford, E. H. Crush, W. F. Farrand, W. M. Gray, T. B. C. Harris, E. J. S. Hewitt, L. B. Holloway, P. N. Hulme, A. MacLean, T. F. Mason, J. McWilliams, R. E. Palmer, A. Rauch and L. N. White.

LIBRARY.

The following additions have been made to the Library since January 1, 1933.

AMERICAN ACADEMY OF ARTS AND SCIENCES. Vol. 67, No. 11. The Eclipsing Binaries RW Aræ and SW Ophinchi. M. B. Shapley.
—— Vol. 67, No. 12. The Purification and Physical Properties of Organic Compounds. 1. The Interpretation of Time-Temperature Curves in Freezing Point Determinations and as a Criterion of Purity. E. L. Skau.

AMERICAN INSTITUTE OF MINING AND METALLURGICAL ENGINEERS. Tech. Publ. 503. A New Contribution to Subsurface Studies by Means of Electrical Measurements in Drill Holes. C. and M. Schlumberger and E. G. Leonardon.

BRITISH STANDARDS INSTITUTION. B.S.S. 52–1933. Bayonet Lamp Caps and Metal-Cased Bayonet Lampholders.
—— B.S.S. 109–1933. Air-Break Knife Switches and Laminated Brush Switches.
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—— B.S.S. 479–1933. Coal Tar Naphthas.

DIESEL ENGINE USERS' ASSOCIATION. The Rating of Diesel and other Engines. W. A. Tookey.
—— Report on Heavy-Oil Engine Working Costs.

EVANS, ELLIOTT A. Lubricating and Allied Oils. 2nd Edition. Fuel Research Board.
—— Tech. Paper 35. Low Temperature Carbonisation. Narrow Brick Retorts at the Fuel Research Station. J. Fraser Shaw.
—— Report for the year ended March 31, 1932.

GEOLOGICAL SURVEY OF ENGLAND AND WALES. The Geology of the South Wales Coalfield. Pt. V. Merthyr Tydfil. A. Strahan, W. Gibson and T. C. Cantrill. (2nd Edition).

INTERNATIONALEN GEOLOGEN UND MINERALOGEN KALENDAR, 1933-34.

LOUISIANA DEPT. OF CONSERVATION. Bull. 22. General Minerals. Bulletin. 1933.

SOCIETY OF CHEMICAL INDUSTRY. Applied Chemistry Reports. Vol. XVII., 1932.

U.S. BUREAU OF MINES. Bull. 351. Mining Petroleum by Underground Methods. G. S. Rice.
—— Rept. Invest. 3197. Composition of the Fractions of Primary and High-Temperature Tar. E. B. Kester and W. D. Pohle.
—— Tech. Paper 543. Comparison of Small and Large Scale Experimental Carbonizing Apparatus. A. C. Fieldner, J. D. Davis, E. B. Kester, W. A. Selvig, D. A. Reynold and F. W. Jeurg.

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WORLD PETROLEUM CONGRESS,
London, July 19th to 25th, 1933.

Arrangements are now well in hand for the World Petroleum Congress, which is being organised by the Institution of Petroleum Technologists.

The meetings of the Congress will be held at the Imperial College of Science and Technology, South Kensington, and a Programme giving particulars of the subjects to be discussed is being circulated with this issue of the Journal.

Membership of the Congress is open to all persons interested in the Petroleum Industry. The Membership Fee is 10s., and those wishing to join are requested to fill in the Membership Form included with the Programme and return this to the Secretary, together with a remittance.

Apart from the fact that only Members of the Congress will be permitted to attend the meetings and other functions connected with the Congress, they will have the privilege of purchasing preprints of the various papers, and the Report of the Proceedings to be published after the Congress, at a reduced rate.

THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

THE ONE HUNDRED AND FORTY-EIGHTH GENERAL MEETING of the Institution of Petroleum Technologists was held at the House of the Royal Society of Arts, John Street, London, W.C. 2, on Tuesday, February 14th, 1933, Mr. J. KEWLEY, President, in the Chair.

The Secretary read the names of candidates nominated for election, and the following list of members elected :—

As Members.—Frank Nettleton Harrup, William Wade.

As Transfer to Member.—Harold Montague Sale.

As Associate Members.—Eric Cyril Cosserat, Keith William Patterson, Mervyn Aleck ap Rhys Pryce, John Westbury.

As Student.—Jacon Jan Leendertse.

As Associate.—Rupert Godfrey de Forembre.

The President said he desired to say a few words about the impending Petroleum Congress. This Congress was taking place between July 19th and 25th, and was being held in South Kensington at the Imperial College of Science and Technology. As far as it had been possible to judge from communications with foreign countries, the interest in the Congress was going to be really widespread. He hoped at the next meeting of the Institution to be able to give much more detailed information,

The Papers Committee had got out its programme in detail. A definite movement had been put on foot for the getting together of the necessary papers, which would be of a general character and which would deal with subjects which had given rise to a good deal of discussion and in regard to which, it was hoped, the Congress would be able to do something to settle on an international basis.

The membership fee of the Congress would be 10s. There seemed to be a little misunderstanding as to what that represented. The fee of 10s. represented membership of the Congress. It need only be paid by those who were attending the Congress, because it was only those who attended the Congress who would get anything for their 10s. It represented, really, a contribution towards the expenses of the Congress. One would get for this the use of the committee rooms, all the general information which was published, free use of various facilities, the privilege of attend-

ing and hearing all the papers, and a season ticket to the Exhibition.

There would be no discrimination made between members of the Institution and non-members with regard to membership of the Congress. The members of the Institution could have no justification or right to expect that they should get better terms than a person who was not a member of the Institution. The Institution was only acting as an agent for organising this world conference. It was costing the Institution nothing, as the funds had been subscribed by the various oil companies. It would not be possible to supply the transactions of the Conference free to members.

All the papers which were to be presented would be printed first of all, and would be available to all members of the Congress at a nominal fee—6d. a copy, or something of that sort, so that a member could get some time beforehand any paper in which he was interested. A résumé would be presented at the meetings and the papers would only be discussed.

With regard to the Exhibition which was being held at the same time, the Institution had no financial or other interest, or responsibility, for this Exhibition. The two events were taking place at the same time, and obviously a successful Exhibition and a successful Congress would be of mutual assistance to each other. The Institution therefore would extend to the Exhibition its sympathetic interest and support, and would be as helpful as it could be in the way of assisting it in educational directions, or the like.

The President then introduced Mr. David Allan, of Price's Patent Candle Company, Limited, who was going to read a paper on the history of the manufacture of candles. The members had been addressed last year by Mr. Allan's brother, who had given them a very interesting lecture on the subject of the refining of paraffin wax. The present Mr. Allan was an equally great authority on the subject of candles. As this was a subject which had never yet been brought forward before the Institution during its life of 20 years, and as it was a subject which very few persons knew much about, he was going to reverse the order of affairs, and instead of asking the lecturer to cut his paper as short as possible in order to allow a long time for discussion, he was going to ask him to read the paper in fairly full detail and limit the time for discussion.

The following paper was then read and was illustrated by means of specimens of candles, models of moulding machines, etc.

Candles and Candlemaking.

By DAVID ALLAN, F.I.C., A.R.T.C. (Member).

A CANDLE may be defined as a cylinder of fat or wax surrounding a fibrous core or wick.

It is, perhaps, the only form of illumination which is completely self-contained. It does not, like oil, require a container, like gas, a pipe to supply, or like electricity, a wire. It is on this account that the appeal of the candle is obvious.

It may be thought that the candle-making industry, if not actually dead, is very nearly so, but I will quote figures later which show that the world candle consumption is a very considerable item, and, further, that the consumption in Great Britain is also still very substantial.

The early history is lost in obscurity. The first type was undoubtedly a piece of pine or other resinous wood, so that the man who discovered the method of making a fire can be regarded as the discoverer of the candle.

The word is mentioned several times in the Bible, but it seems fairly certain that the earlier references, at any rate, relate to lamps and "candlestick" to the support for the lamps.

When the art of making candles was first discovered is not known, but they are mentioned by several old Greek and Roman writers. In many cases it is not clear whether torches are meant, but Pliny states that the pith of brittle rushes which grow in marshy districts was used for making "watch-candles" and funeral lights to burn by dead bodies while lying above the ground, but he does not say anything as to the nature of the fat or wax used.

That the art was known to the early Romans is evident, for Apuleius in a kind of novel called "The Golden Ass," written about the middle of the second century A.D. (Metama IV.), mentions that "at a noise in the night the household runs in with torches, lamps, tallow candles and wax tapers (or candles)." The tallow candles were probably rushlights, and the wax candles similar, except that beeswax was used instead of tallow. There are also specimens of Roman candlesticks in the British Museum.

Beckmann ("History of Invention," Bohn's Ed. II., 174) has recorded that the Emperor Constantine, at the beginning of the 4th century, caused the city of Constantinople to be illuminated with lamps and wax candles on Easter Eve.

There is also at the British Museum a fragment of a candle found in Vaison, near Orange, which is supposed to have been made about the 1st century A.D., and a chandler's apparatus was found in the ruins of Herculaneum.

The earliest type of wick was probably a thin piece of porous wood, such as is used even at the present day by the natives in China. These were dipped in the fat or wax, and in some cases rolled into shape. This was probably followed by a roll of papyrus, somewhat similar to the roll of paper which the Japanese use at the present day for some of their Rickshaw candles. Oakum and linen were also used, as the cultivation of flax appears to have been known from very early times. Later on, the common rush and cotton were used.

Gilbert White, in his "History of Selborne, 1789," gives an account of the method of making rush candles practised in Hampshire at that time. He says: "The proper species of rush for this purpose seems to be the Juncus conglomeratus, or common soft rush, found in most moist pastures. These are in the best condition in the height of summer and early autumn. As soon as they are cut they must be thrown into water and kept there, for otherwise they will dry and shrink and the peel will not run. At first a person would find it no easy matter to divest a rush of its peel or rind, so as to leave one regular, narrow, even rib, from top to bottom, that may support the pith; but this, like other feats, soon becomes familiar, even to children. When the rushes are thus far prepared, they must lie out on the grass to be bleached and take the dew for some nights, and afterwards be dried in the sun. Some skill is required in dipping them in the scalding fat or grease, but this knack also is to be attained by practice." He says that 1600 rushes, weighing 1 lb., are coated with 6 lb. of tallow, so that 228 lights weigh 1 lb. and cost a little over 5d. From this it will be gathered that the light would only be a short-lived one. The making of these was general in country districts; they were made in the home from locally collected rushes and domestic tallow supplies.

The torch and its modifications—the link and flambeau—consisting of rope saturated with pitch or wax had been in use for centuries. Even as late as the 18th century they were carried about in the streets of London, and about fifty years ago extinguishers for these were still to be seen in the doorways of old London mansions, but whether there are any in existence at the present day the author cannot say.

Although tallow dip candles had been known from very early times, they do not appear to have been in common use until the beginning of the 17th century, and the makers were a separate class from the wax chandlers. When the rush wick was superseded by cotton is not known. Candlesticks to take these candles were well known, and later on they were fitted with a spike on which the butt of the candle was pushed. The same idea is in

use in Eastern countries to-day, and the candles are specially made hollow at the base to fit over the spike.

The Sieur De Brez is said to have introduced the art of moulding candles in the 15th century, but very little progress was made, owing to the difficulties encountered in moulding beeswax, and the fact that only the harder qualities of tallow could be used.

Beeswax candles had been used for centuries but were always made by hand, as indeed many of them are still. They were made by pouring the melted beeswax over wicks suspended over a bowl until a sufficient thickness was obtained, when they were taken down and rolled into shape on a smooth marble slab and cut to sizes.

Asser mentioned that King Alfred had wax candles made so that six candles weighed equal to 72 pence. These were of equal length and had 12 divisions marked on them, and the six when lit in succession burnt exactly 24 hours. Fosbrooke, according to the "Encyclopædia of Antiquities," stated that in the Middle Ages wax candles were made of various sizes, some very small and others weighing as much as 50 lb. He also states they were made in moulds, and that the wicks were formed of twisted tow.

These wax candles were mainly used by the churches and wealthier classes as they were, of course, much dearer than tallow candles. The use in churches dates back to very early in the Christian era, and to-day certain candles used in Roman Catholic churches have to be made either wholly of beeswax, or contain a definite percentage of that material, usually from 25 to 65 per cent, sometimes 75 or 95 per cent.

Venice has been regarded as the home of the wax candle trade, but it is evident that it was an important one in London in the 15th century, for the Wax Chandlers' Company was founded in 1484, and the Tallow Chandlers' Company a little later. They are twentieth and twenty-first in order of civic precedence of the City Companies.

In London the wax chandlers lived at the eastern end of what is now known as Cannon Street, and the present name of that street is a corruption of Candlewych Street.

There was very little improvement in the manufacture of candles until Chevreul published his classical researches on the nature of the fats between 1813-23. Until then the only materials used in Britain and on the continent of Europe were tallow, beeswax and a little spermaceti, although the Chinese used insect wax and vegetable tallow. In 1799 a patent was taken out by William Bolts for improving the form and quality of candles by the use of pressure to obtain the harder stearine from tallow—this stearine, of course, is the glyceride and not the fatty acid which is the usual

material for candles of that name to-day. He also describes a solid candle with a short wick, which was placed in a holder and kept pressed on the candle by a spring, or the candle was placed in a tube and pressed against the wick by a spiral spring; evidently the forerunner of spring-tube lamps and candles used now.

The most important improvement, however, was the introduction of plaited wick by Cambacères about 1820, which did away with the necessity for snuffing, although these wicks are not suitable for tallow candles which require a very loose wick on account of the low melting point of the material. Previously only twisted wicks, usually of cotton yarn, had been used for all candles.

Although Chevreul published his collected researches in 1823, it was two years later before he and Gay-Lussac took out a patent for the manufacture of candles from the fatty acids. They built a factory to carry out the process, but mainly owing to high costs the undertaking was not a commercial success. Then another Frenchman—De Milly—modified the process by substituting lime for the expensive caustic soda and potash, which Chevreul and Gay-Lussac had used, and this process was worked on a commercial scale in 1832. In the year 1833 25 tons of stearic candles were made in Paris by MM. de Milly and Motard, and were marketed at 1s. 8½d. per lb. retail.

In 1831 candle manufacture was set free from Excise supervision and from the duty which had been 3½d. per lb. on wax and spermaceti candles, and 1d. per lb. on tallow candles. These duties realised in 1829 £489,059.

In 1829 James Soames took out a patent for separating coconut oil into its solid and liquid parts, and this was sold to Edward Price and Company, who worked it on a fairly large scale and made candles with the stearine, but as the candles required snuffing they were not used very extensively, although the use of coconut stearine was continued for many years mixed with fatty acids or stearines, and Messrs. E. Price and Company set up mills in Ceylon to extract coconut to supply the London factory.

Here it may be opportune to note that Edward Price and Company—the predecessors of Price's Patent Candle Company—was only a business name adopted by two City merchants—Messrs. William Wilson and Lancaster—for the purpose of working the above patent.

It is also of interest to note that in 1836 the use of hard acids from palm oil produced by pressing the neutral oil and saponifying the cake had been patented by Messrs. Hempel and Blundell, and some quantity was produced and used, but the dark colour told against its general adoption.

The French process for making stearic acid was introduced into

England about 1835, and was worked by the company. About the same time they first used coconut fibre matting instead of sail-cloth in pressing the coconut oil, and this material has been used ever since for this purpose.

In 1836 the plaited wick patented by Cambacères was made at Ripley by Mr. Thomas Topham.

In 1840, on the occasion of Queen Victoria's marriage, Price & Co. brought out their composite candles for use in the window illuminations, using this wick. These candles were made of a mixture of stearic acid and coconut stearine, and as they did not require snuffing and were cheaper than all stearic acid candles, they became very popular. They were sold at 1s. per lb. The reason why stearic acid candles—or stearine candles as they are now universally called—were not so popular, will be told later in the paper.

In the same year Gwynne took out his patent for distilling fatty bodies in a vacuum apparatus, and also for distilling fatty acids and soaps under atmospheric pressure. Two years later Edward Price and Company and W. C. Jones patented a method for distilling coconut oil acids and also after saponification with lime. The candles made from the first product were not satisfactory, owing to the choking vapour given off when the flame was extinguished, and they were never marketed. But the latter part of the process gave very good candles which, however, were too costly.

A few months afterwards—December 8th, 1842—Wilson and Jones obtained a patent for decomposing fats with sulphuric acid and distilling the product in a current of steam. This was the first successful process for distillation of fatty acids, and with slight modifications, such as the use of superheated steam patented by Gwynne and Wilson in 1844, is one of the chief processes used to-day. They followed this with further patents on a high temperature acid treatment for production of solid from liquid acids (iso-oleic from oleic). This was in contrast to a process by Frèmy in 1836 for treatment at low temperature with half its weight of sulphuric acid. Such a cold process had been patented in England in 1840 by Clark and Gwynne.

In the new process 33 per cent. acid was used, but this was subsequently modified to 3½ per cent., a figure which is used when such a process is necessary to-day.

Candles were made of a mixture of these fatty acids and coconut stearine, and were those already mentioned as being sold on the occasion of Queen Victoria's marriage.

The invention of the acidification and distillation process permitted of the use of a considerable range of raw materials, such as bone fat, skin fat, recovered greases and palm oil. It may be

noted that the importation of palm oil to England rose from 19,800 tons in 1840 to about 50,000 in 1871.

Shortly after the distillation process was adopted, pressing of the distillate was carried out for production of stearine for better class candles.

In the early days these stearine candles were moulded by pouring the hot liquids into moulds. To overcome the crystalline appearance on the surface, arsenic was added—in what quantity we cannot trace—but it was not long before this practice was dropped, as it seriously affected the popularity of these candles. There is no record of any fatal effects, but it was said that candles giving off arsenic did affect the crew of one Arctic expedition. To overcome the difficulty which arsenic was used to obviate, the practice in use up to the present time was adopted. This is to stir the stearine until it is in a creamy consistency before moulding and, by filling into a warm mould, absence of surface marks is obtained with much improved snap.

The oleic acid separated in the early days of stearine making was used in burning oils, but very soon a new use was discovered for it which persists until to-day.

This development in the use of oleic acid permitted the rapid extension of the production of stearic acid for candle-making, which otherwise might have been considerably hampered by the large amount of oleic acid which is produced at the same time as stearine, and as a result in 1854 the company opened near Liverpool at Bromborough Pool a large factory to work their several patents, with palm oil as their chief raw material.

A year later, 1855, was produced glycerin. This was the outcome of the work of Mr. G. F. Wilson, who was the founder of the stearine industry in this country, as the result of his previous discovery that neutral fats could, by distillation with superheated steam alone, be broken up into fatty acids and glycerin. The natural result followed of distillation of glycerin alone, giving the first chemically pure glycerin. This is another contribution of candle-making to science and industry.

About this time in 1850 Mr. James Young obtained his patent for the production of hydrocarbons by distillation of coal, etc.

In 1853 and in following years Mr. Warren de la Rue was granted patents for working Burmese or Rangoon petroleum. The company secured the rights of working these and sent agents to obtain supplies.

By 1857 regular supplies were being obtained. Many difficulties were at first experienced in converting laboratory working to commercial success, but after some time excellent products were

obtained, and it is recorded that over 10,000 gallons of burning oil were shipped to New York in 1859; but the discovery of American supplies just after this put a stop to such shipments and seriously upset the calculations that had been made of the value of the Rangoon material and the importation had to be stopped.

While the Rangoon petroleum was being worked it was distilled, as were the fatty acids, in superheated steam for which a patent was taken out by Wilson in 1849. The products made were light oil, burning oil, spindle oil and paraffin wax. It may be of interest to know that the latter product was valued at that time at £200 per ton, but owing to the American discovery that value did not long remain.

The spindle oil was, after some difficulty, introduced into spindle oils for the Lancashire cotton industry mixed with sperm and other fatty oils. This is the first use of mineral oil for that purpose. While the Rangoon oil proved unprofitable for the reasons stated, it was the beginning of an extensive lubricating business.

With the advent of Scotch paraffin and that from America, this material became a very important one to the candle-maker, and paraffin refining grew to very large dimensions in Great Britain. The refining was done first by crystallising with naphtha and hydraulic pressing. This process was carried on until 1871, when Mr. J. Hodges made his discovery which developed to the present-day sweating process, and subsequently about 1890 the company was the largest refiner of paraffin wax in the world.

Mr. Hodges' first trial of sweating was done on a block which he allowed to rest on sand in a warm place. He was led to this through having been aware of a process which was proposed for refining wax by kneading the wax in warm water.

As the result of Mr. Hodges' trial numerous experiments were carried out in the laboratory in 1871 to determine the best conditions to work under, and it was found that coir matting was a suitable support for the blocks, and that slow sweating at as low a temperature as possible was more efficient than short time at a higher temperature. The early sweating was done on blocks which had been cooled and were sweated between coir mats on inclined heated plates in small ovens. Later this laborious process was changed to trays in which the paraffin was cooled by means of water circulating through pipes in the mass as well as by atmospheric cooling, and the sweating was produced by circulation of hot water through the same system. The unit was gradually increased until the ovens were capable of dealing with 50—60 tons per batch in a 48 to 60 hrs. cycle, depending on weather conditions.

There is no need to say more on the sweating question, as more

recent developments were dealt with in a paper read here two years ago.*

The wax mostly dealt with from 1890 to 1922 was American Yellow Scale 122/4° A.T., which was separated into 118/20° and 125/7° E.T. with at times producing 130° E.T. At first Eastern wax with its higher S.P. was expensive, and not so plentiful, so that stearine was still an important factor in candle-making, particularly in candles for hot climates or which had to stand passing through tropical seas.

If the amount of light per unit weight were the most important factor, then paraffin wax is the most suitable candle material. Compared with stearine and the standard spermaceti candle, the relative amount of light given for a consumption of 120 grains per hour of each of these materials is :—

Stearine 0·84, spermaceti 1·0, paraffin 1·14, and the light from paraffin stearine mixtures can be calculated from these figures.

For certain markets pure stearines were, and still are, demanded, as, unlike paraffin, stearine candles do not bend much below their melting point.

Stearine can be added to paraffin to remedy this to a degree, but so far no mixed paraffin-stearine candle has given the same stability to heat as pure stearine, though there are few purposes only for which a satisfactory mixed candle cannot be produced with the aid of 135/40 M.P. wax and stearine. Of course, S.P. of paraffin wax is not the sole criterion on which a refined wax is valued for candle-making. There is no strict relationship between S.P. and bending point. Waxes of different origin show different stability at the same S.P., and the difference can be measured in terms of stearine required to bring it up to the same figure.

In the earlier part of this paper mention is made of the production of iso-oleic acid by acidification. This is a satisfactory constituent within certain limits in stearine candles, but in mixed candles it may be detrimental. If opacity of the candle is the important point, then it is satisfactory, but if stability is wanted it is of little or no value. For certain mixed candles, therefore, it is better to use stearine produced in such a way that iso-oleic acid is present in as small quantities as possible.

The making of stearine is now carried on mainly without the acidification process, and this method of production happens to be better for stearine for many other purposes, which are now as important an outlet as the candle industry.

Such uses as in the rubber industry, cosmetics, soaps and stearates absorb an increasing tonnage of what was formerly practically

* Apparatus for the Sweating of Paraffin Wax. H. L. Allan. *J. Inst. Petr. Techn.*, 1931, XVII. 656–668.

wholly a candle material. A brief summary of the process of making stearine may be of interest.

In the early days fats were split by sulphuric acid, then later by lime saponification. Between 1872–76 the autoclave was introduced to do the saponification under pressure of 120–130 lb. with 2 per cent. lime. This was first done by De Milly in 1855. Later zinc oxide was used, also magnesium oxide; 1 per cent. of the latter is commonly used and about 6-hrs. pressure is given. Change of water is made, usually after 3 hrs., as the removal of the glycerin water allows the saponification to be carried further in the second stage. The batch is blown out and the so-called sweet water separated, and if recovery is worth while the charge is washed to remove the last of the glycerine. The soap is decomposed with acid and washed and settled.

For quite a long time this was the only process used, but in 1898 Twitchell found that the sulpho-aromatic-fatty compounds had the property of splitting fats on boiling with steam and a small percentage of sulphuric acid, into fatty acids and glycerin. This process is now very extensively used, particularly where distillation is to follow, as the process tends to darken the fatty acids.

Many reagents have been proposed and used as splitting agents; one which is now used extensively is the sulpho-naphthenic acid derived from petroleum refining. Materials of this nature have superseded to a very large extent the naphthalene-sulpho-fatty compound of Twitchell. They were first discovered and their use for this purpose was patented by Petroff.

The Twitchelling process is, in brief, as follows :—

Cleansing of the raw material where necessary. This is carried out on the well-settled material at about 180° F. by agitating, generally with air, with a quantity of 80 per cent. sulphuric acid, varying according to the raw material, but averaging about 1 per cent. After thorough settling, which is sometimes followed by a water wash, the fat goes forward for splitting.

The splitting is done in wooden tubs of suitable size, say, up to 50 tons total capacity.

The charge is given about 1 per cent. reagent on the fat and ½ to 1 per cent. of sulphuric acid and sufficient water to give about 20 per cent. glycerin solution at the end of the first boil.

The boiling is by open steam and varies in time from 12 hrs. upwards, depending on the raw material, but is regulated by the split, which should be about 88 per cent., before the water is taken off.

Fresh water, about 15 to 20 per cent. on the fat, is added with 0·25 to 0·5 per cent. acid, and boiling continued until split is 92 to 95 per cent., when a further change is made.

Rather less water is added the next time, and split completed to 95 per cent. up to 99 per cent., depending on the raw material being dealt with.

Having got your fatty acids, or 95–97 per cent. fatty acid, the next process is distillation. The original superheated steam distillation of Wilson still holds favour with many stearine makers. Vacuum stills are also in use, particularly for distillation of cheap fats for soap-making purposes, but there are certain features of the old atmospheric steam distillation which are beneficial for the stearine-making process.

The distillation in the latter type of still is carried out in stills of various sizes and construction. We favour copper stills of capacity to take 9 tons, others, smaller stills of iron. In either case fire heat is supplied and superheated steam. We consider this is best supplied by a superheater apart from the still, but what is necessary for copper is not necessary, though probably more efficient, for iron. The steam is superheated to 800–850° F. and the pressure maintained at about 5 lb.

Condensation is by atmospheric condensers of copper with water-cooled coils at the bottom of the bend which joins the adjacent legs of the atmospheric condenser. Finally, the volatile vapours and steam are condensed at the end by a jet of water, or, if desired, by a cooling element of other type.

The distillation is carried on as long as the colour of the distillate is good, which depends on the split of the fatty acids, but runs to close on 90 per cent. with good materials. The bottoms may be blown out and resplit for distillation.

The distillate is then pressed either first cold and then hot or directly hot pressed, depending on the nature of the material. Fractionation at the stills is also carried out to vary the crystal of the stearine when desired, as certain proportions of palmitic acid to stearic acid give the best stearine for moulding stearine candles. The oleic acid is pressed out from the cold presses, and may be further chilled and filtered before it is converted into material suitable for wool lubrication.

That, very briefly, is the present stearine-making process, omitting details which are not of interest here.

It will be obvious that up to about 1840 the scope for serious modification in candle-making machinery was limited by the quality of materials which could be obtained cheaply enough for common use. However, as soon as materials harder than tallow became available, the development in moulding became possible.

Moulding of candles was not new at this time, although more modern than dips, the introduction as already mentioned being ascribed to Sieur de Brez, of Paris, some time in the fifteenth

century. The exact form of his frame is not known, but hand frames were in common use well past the middle of the nineteenth century.

These were simply a set of moulds in wooden frame with arrangements for centring the wick top and bottom. The cooling is merely atmospheric and of necessity is a somewhat slow process.

It is in a frame of this type that candles used as standard for illumination were made, and in fact are still made. The material used is spermaceti with a small addition of beeswax to help moulding, and the wick used is regulated to give 120 grains per hour consumption. The light from this is the standard of 1 candle-power.

The first use of steam and water for heating and cooling candle moulds was patented in 1801 by Thomas Binns, of Marylebone. The only feature of his machine which we will draw attention to was the fixing of the moulds in a steam-tight box into which water and steam were alternately introduced. He claimed with truth :—

"The great advantage of making mould candles by applying steam and cold water in succession round the outside of the moulds, makes them more expeditiously and beautifully than heretofore, as there is no necessity for waiting until they are cooled by the air, and when made will have a better gloss than common candles. These moulds are particularly adapted to the making of wax candles, which have never before been made perfectly in moulds on account of the difficulty of keeping the moulds warm while the wax was poured in, and from the impracticability of drawing them out when made, which is entirely remedied by introducing steam and water in succession round the outside of the moulds."

In 1796 Sampson, of Moorfields, Middlesex, patented a machine which, however, was merely six hand frames shaped to form segments of a circle which could be turned round and filled at the tank of melted material.

In 1832 Wm. Palmer took out a patent for a machine which combined water cooling and a system so that the machine could be emptied of water and turned upside down to get the candles out.

Without going into details, many patents were taken out between 1830 and 1855 by Joseph Morgan, of Manchester, Joseph Tuck, of London, Palmer and others for various improvements, such as ejecting the candles, centring the wick by means of a clamp holding the candles, etc. The prototype of the candle machine of to-day was an American invention by John Stainthorp, of Buffalo, N.Y., who was said to be a Yorkshireman familiar with the existing form in this country. He combined many of the known improvements in one machine.

A few months later a further American patent was taken out by Willis Humiston which had some points of difference, such as an

arrangement to give some longitudinal play to the rams in order that a sharp blow may be communicated to the tip and start the candles out of the moulds. He also claimed a slight difference in method of holding the clamped candles to centre the wick for the next batch.

Between the two it can be said that the modern candle machine is made up.

In 1857 a method of ejection by means of compressed air was patented by Mr. E. A. Cowper, who was consulting engineer to Price's Company, and was used for many years with success.

The modern machines are now produced on very similar lines in all countries with differences in size, etc., to meet special requirements. The important points in machines are, method of fixing moulds, ease of cleaning tank, should water be dirty or corrosive, and ease of handling.

Self-fitting ends were first patented in 1861 by Mr. J. L. Field. These have either to be moulded with caps which are removed after winding up or with a split cap to open and let out the candle. We find the former to be preferred.

Modern candle-making is carried out in frames of the type shown, which vary mainly only in the number of pipes they contain.

The larger the frame the more wick is required under it, but there is a maximum height, beyond which it is not possible to work a machine easily.

This led to the adoption of a system having the wicks in a room underneath with a separate attendant to look after the supply. It is doubtful if such a layout is worth the extra cost of construction, and we have found it possible and economical to work 518 pipe machines without having to resort to this sytem.

Regarding the procedure in candle-making at the present time, the wick is, as you see, on spools under the machine, and at first wicking is drawn up through the ram by means of a piecing stick which resembles a crochet needle. The machine is filled with material, then cooled, the top scraped off with a sharp spud or knife, and the candles wound up into the clamp where they are held. If the candles are clean the machine is ready to begin moulding candles.

The exact procedure depends on the nature of the candle material and is related to the S.P. of the mixture, temperature of cooling water and atmospheric conditions, the object being to adjust the temperature of filling, so that the material is not chilled too rapidly when filled and so leave marks or airholes. The necessary experience is soon acquired and the temperature of machine and material to be moulded are kept as regular as possible. In the case of the material, particularly with high S.P. wax, the use of jacket

pans is necessary. These are preferably of aluminium or enamelled iron, and supplied with steam at about 5–10 lb.

The hot material from these jacket pans is filled by means of a pail, sufficient material being used to leave a layer of about ¼ in. on top bed plate when cold.

We are aware that a system for running material direct from storage tanks into the candle frames is worked, but we consider this practice has many disadvantages unless under special circumstances.

The cold water is then turned on and the candles set in the moulds. When sufficiently set the candles in the clamp are cut off by means of a sharp knife run along under the clamp. These are then thrown from the clamp on to the packing bench or other receiver. The top material is scraped off, using a sharp spud, and this material is either returned to the jacket pan or reboiled with a fresh batch. The candles left in the moulds are then wound up and the cycle goes on.

In moulding stearine candles, as mentioned before, the material is filled cooled to a creamy consistency, and cold water must not be used for cooling as this may crack or otherwise damage the candle. The moulding of a stearine candle thus takes much longer than a paraffin mixture.

As regard the moulding of paraffins, all paraffins do not behave in the same manner, and frequently, in the absence of stearine, additions have to be made to prevent mottle or to prevent sticking. Materials to prevent mottle are numerous. Perhaps the most common is carnauba wax. Lead stearate is sometime used, also montan wax and its products. Mottle has also been prevented by removal of the dissolved air by means of steam just before filling, but the process has disadvantages.

Small quantities of stearine prevent mottle and assist moulding, provided the stearine is of suitable quality. Stearine containing considerable proportions of iso-oleic acid is useless for this purpose in small quantities, but in larger amounts is satisfactory.

In the case of sticky waxes oil may be added, and for this purpose coconut oil is useful, but it may produce mottle.

Some anti-mottle agents, such as lead stearate, cause stickiness and difficulty in ejection.

For a number of years the benefits obtained by addition of stearine to paraffin for use in overseas markets has been widely recognised, and opacity has been looked on as an indication of quality. Such an appearance cannot now be taken to indicate the presence of stearine. Opacifiers have been discovered, the addition of a small proportion of which gives the appearance of a composite paraffin stearine candle.

The addition of 0·2 per cent., 0·4 per cent., 0·6 per cent. of opacifier give the appearance of 10 per cent., 20 per cent., 30 per cent. stearine respectively.

With the advent of supplies of Eastern high S.P. waxes the same standard of stability and appearance can be kept up with less stearine, by the addition of opacifier.

It may be added that the opacity is not always apparent when the candles are moulded, and it may take some days before it is fully developed.

No mention has so far been made of ozokerite, which is obtained from the natural earth wax. In 1870 Field patented the use of the distilled ozokerite for candle making.

Ceresin is the natural occurring ozokerite purified by chemical means, but owing to cost the use is very limited, and it is not as good a candle material as paraffin wax.

Of recent years there has appeared, particularly in Japan, considerable quantities of stearine made from hydrogenated Japan fish oil, known there as "stearine RO," also hydrogenated sperm body oil, a neutral wax known as "kokaro." They are of a different nature to the usual stearines on the market, in that they contain some very low M.P. acids, which are undesirable. The sperm product does not give either the same opacity or stability to a mixed candle as ordinary stearines, but, particularly in Japan, where they can be produced cheaply, they are a serious competitor to paraffin wax.

It might be mentioned here that two other methods of making candles have been tried, but as far as we know only one of these is being worked. This is the method of making as in taper making, where the wax coating is built up continously by passing through a bath and dies till the required thickness is obtained. The candles are cut off and tipped by a machine and are also given some polish.

The finished article would not find favour in this country, but is used in Poland. The cost of the method in this country would not, we estimate, compare favourably with the machine, and there is no comparison between the finish of the two.

The other method which we cannot trace as having been successful, is the method of extrusion. It is possible that with certain waxes the method is feasible, but we feel sure it is not a method which can be adapted to all waxes.

As is well known, candles are made in a large variety of sizes, shapes and colours.

Next to nothing has so far been said of the wick, although this is a very important part of the candle, and much depends upon its construction and treatment how the candle will behave in use.

As mentioned before, the plaiting of wick was first invented by Cambacères. Before this time it was necessary to snuff the candle and remove the unburnt wick as it became too long and caused much smoke.

By plaiting the wick the end was kept at the outside of the flame, and by suitable treatment it was possible to keep the end clean and red.

Exactly why the wick should curve in the particular way it does the author has never been able to find out, but the fact remains that the wick will only remain the one way when burning, and if packed with the wick bent over the wrong way, as it must of necessity often be, it has to burn about ¾ in. of candle before it gets the right way. During this period it has to pass through the flame and is in danger of forming a "cauliflower." The length of timé it takes depends on the shape of tip and it may be the top was originally made with this in mind, as the tips in use are generally the best shape for this purpose, consistent with sufficient strength to remain unbroken.

The wick is usually composed of three groups of an equal number of strands of fine cotton yarn, the most common being 3/10s and 3/12s, which are plaited together. This was for a time the only wick in use, but it was found about 60 years or so ago that coarser yarn could be used if the wick was made up of three ends twisted and three of these plaited together. This gave an economy in weight of yarn for the same consumption and also a cheaper yarn could be used.

The use of this type of wick has of recent years been extended beyond this company's use, who for many years were the sole consumer.

After the wick has been plaited it is treated to the caustic boil and bleaching usual to cotton, washed and centrifuged. After this it is treated with solution to assist clean burning.

The composition of solutions are many and varied. The following salts being the chief used : borax, ammonium sulphate, ammonium chloride, potassium nitrate, potassium chloride and ammonium phosphate.

The selection of a solution for this purpose is very important and many factors have to be kept in mind. The quality of the candle to be burnt is not unimportant or it any special conditions of burning have to be met. It is, however, hardly necessary here to enter any further into details of this.

Closely associated with candle-making has been the making of night lights and tapers.

Night lights were originally sold as mortars or death lights, and were used to light up the death chamber. In 1842 we find the

production of 63½ dozen lb. for the month of January. This was apparently the first time these were produced by this company. In 1849 they took over Childs, and the earliest form was made by filling by hand from a vessel like a coffee pot into a case provided with a wick and sustainer; these cases were for a time made of thin wood shavings, owing to the duty on paper.

Later the hand filling was replaced by machines for moulding, on similar principle to the candle machine, except that in place of a wick there is a wire to form the hole for the wick, and this mould is placed on to the wick in the case.

The wick is made into a taper and cut in short lengths and fixed with a sustainer in the bottom of the case. The light has the hole completed by means of a hot piercer, and is then put on to the wick.

The regulation of the wick and the material for night lights is a matter that requires constant supervision, if a regular burning time is to be obtained. The S.P. of the wax is also adjusted to the climate in which they are intended to be used. The S.P. has to be high enough to prevent the lights becoming misshapen and low enough to be melted completely at the lowest temperature they are likely to be used. This is an ideal which is not always capable of attainment, but is usually so under average conditions. The rate of consumption is low, being about 40 grains per hr., as against about three times that for a candle.

The production of night lights is still a very important business in this country at least.

Special forms of lights are made for burning in glasses. Paper-cased lights to be used without water, as against the original with water, are in increasing demand. These are all practically pure paraffin wax.

A special form is also made from neutral fatty material, in which rush wicks are used. These give a good soft light and are used for decorative purposes.

Tapers are still made, but in this case the proportion of cotton is high, and no plaiting is necessary. The percentage of cotton varies with the thickness of taper from 12 per cent. to 18 per cent. The wax used is mainly paraffin, with varying quantities of stearine or beeswax, according to quality.

The making is simple. The cotton, in lengths of about 1800 yards, is wound from one drum to another through a bath of wax, and through dies till the required thickness and finish is obtained, then cut in lengths and the ends feathered by washing off the wax in hot water in order to make them easier to light at first.

Coming now to the candle production of the world, it is extremely difficult to procure figures which can be considered absolutely reliable for the total tonnage of candles produced and consumed

all over the world, because in such places as China large quantities of candles are made by natives from local raw materials, such as Chinese vegetable tallow, but the total world production, neglecting some of these sources, is certainly well over a quarter of a million tons per annum, and of the individual countries the United Kingdom, including the Irish Free State, is probably the largest individual consumer, with about 45,000 tons per annum.

It is estimated that in the United Kingdom the average size is probably about twelve to 1 lb., which indicates the very large number of candles consumed in this country alone.

With regard to the uses of candles, there are three main uses ; the first is ordinary domestic use, the second is the use for religious purposes, and the third for industrial purposes.

The vast majority of candles used in the United Kingdom are for domestic purposes ; there is also a considerable consumption for religious purposes. In the industrial uses are included mining, boiler-making and other similar uses.

In Eastern countries the quantities used for religious purposes are very large indeed, and in Japan in particular the candle consumption is almost wholly for religious purposes.

It is difficult to obtain an estimate of the total amount of beeswax used in church candles, but the figure runs into many thousands of tons per annum.

Of recent years considerably increased interest has been displayed in the use of candles of various shapes and designs, and in a large range of colours, for decorative purposes, and although the quantity consumed in this way is quite considerable, it is not very significant when considering world tonnages, but it introduces its own problem in obtaining suitable colours which will not interfere with the burning and will be reasonably fast to light.

Taking into account the world-wide trade depression, it is probable that any reduction in paraffin consumption for candle making is only temporary. In any case, the candle-maker still remains a very important consumer of petroleum refiners' products.

DISCUSSION.

The President said the members had had a very instructive and interesting lecture. The subject of candle making was one of which comparatively little was to be read in the literature.

The author had touched lightly on the question of the bending point. He (the President) would like to ask to what the bending point was related ? The author inferred that it might be related to the origin of the wax. The President thought there were two

possibilities. Firstly, it might be related to the degree of fractionation of the wax. Did it depend upon—if one was dealing with, say, a 125 melting point wax—whether the components ranged from 120 to 130, or more or less ? Had anything been done to clear that up ? Secondly, it might be related to the chemical composition of the wax. He thought that very little was known about the chemical nature of these paraffin waxes. There was a general impression perhaps that the paraffin waxes were normal straight-chain hydrocarbons. He himself had been under that impression until recently. A certain amount of work was being done now in the direction of relating the physical properties of these hydrocarbons to their melting points, and several people were working on the structure of paraffin wax by means of X-ray analysis. There was sufficient to show definitely that the ordinary paraffin waxes were not by any means straight-chain hydrocarbons, but that they consisted largely of branched chain hydrocarbons ; and there was some evidence that waxes from different types of crude did definitely display differences in their chemical compositions in the relative proportions of branched and straight-chain hydrocarbons which went to make up the wax as a whole. A more definite answer to this question could certainly be expected later on.

The question of mottling was one of great interest too. Years ago in Borneo he had been engaged in the manufacture of paraffin wax, and had had a great deal of trouble with this question of mottling. He thought it had some relation to the solubility of air in paraffin wax. If paraffin wax was allowed to cool in a vacuum a wax was obtained which was practically absolutely transparent ; if it was cooled in air it was relatively opaque. He thought that during the process of crystallising a good deal of air was liberated from solution, and that the opacity of the wax was largely controlled by the presence of this air between the crystals. Under certain circumstances it was possible to get a wax which contained so much air dissolved in it that in crystallising out large bubbles were obtained. It reminded one of the phenomenon of the spitting of silver.

The question of the stickiness of waxes was very interesting. He had not the remotest idea why certain batches of wax would stick to the moulds whereas others came out quite easily. The addition of a small quantity of oil destroyed the stickiness, but there were objections to the addition of such oil.

Mr. J. Gillespie remarked that it was not often that the members had the advantage of seeing a practical demonstration at a meeting of the Institution. Mr. Allan was to be congratulated on his paper and on his demonstrations, and the members of the

Institution were to be congratulated on having this information at their disposal.

He had been pleased to see that Mr. Allan had started his paper by saying that a candle might be defined as a cylinder. There were other forms of candle which, he thought, were not quite so happy. He had seen a three-cornered one, and to his surprise he had been introduced to a square one about three weeks ago. He felt that the man who wanted a square candle might be classed with the man who desired a blue rose.

Mr. Allan had referred to the link extinguisher. If Mr. Allan would walk down the west side of Berkeley Square, he would see a number of those link extinguishers still existing as an interesting feature of the guard rails of some of the houses there.

Personally he was not in a position to make the technical criticism or amplification of the paper which the President had so ably done that night, but one point did occur to him. Although the matter emerged all through the paper, Mr. Allan had not specifically mentioned the question of development in design of the candle. Mr. Allan had spoken of the flambeau, the torch and the link. In all those three earlier forms the wick occupied a very large proportion indeed of the whole. In the candle as we had it to-day the wick occupied a very small proportion—with greatly improved results in the matter of burning without smoke. Mr. Allan had referred to that peculiar curve which the wick assumed just at the burning point. He had recently had the privilege of discussing with his friend Mr. Pemberton certain questions in connection with candle making, and that point had come up—this curve on the wick always holding the point in the hottest part of the flame so that the wick was reduced and disappeared and snuffing was done away with. Mr. Pemberton had suggested that that very desirable result was secured by the use of the special solution with which the wick was treated before it was put into the candle.

It was sometimes a matter of surprise to him that the candle industry held on with so much success. In these days of intensive illumination the candle still held its own place, even against electricity and gas. The same experience had been noted when coal gas was first introduced. It was then said that the candle industry was doomed, but the reverse was the fact.

The more light to which the users became accustomed in public rooms, the more light they desired in apartments where gas was then considered unsuitable. And so the use of candles grew.

Those—and they were fortunately few in the audience—who could go back to a Victorian dining-room and dinner table lit by candles only would understand him when he said that there was no more beautiful light than candle light.

E. R. Redgrove remarked that he was rather surprised to notice that copper stills were used for distilling the fatty acids. He would have thought that there was a great chance of small amounts of copper salts being formed, which would colour the stearine and, perhaps, affect the burning qualities of the candle. Would the author say whether the presence of copper salts mattered in the final product and whether any steps were taken to prevent their formation.

Mr. T. R. H. Garrett said the extinguishers to which Mr. Allan had referred were still to be seen. He had seen two in Charles Street, W. 1, that day.

With regard to the religious use of candles, and referring especially to beeswax, the Orthodox Church insisted on a high percentage of beeswax in the candles.

He was not at all surprised at the figures given for the consumption of candles in their country, and he thought that the demand would be a long lived one. He always used candles at home for a good reason. They were much cheaper than electric light at 8d. per unit and did not require a capital outlay of £150 to instal, which were the figures he was quoted under the new grid system.

The President said he had been making a little calculation. 45,000 tons of candles (the figure mentioned in the paper) only represented 1,209,600,000 candles in the United Kingdom per year. That was only about 4,000,000 candles a day, which was one candle per ten of the population. There was plenty of scope yet!

Mr. Allan, in reply, said with regard to bending, there was very little known about why one particular wax bent more readily than another. It had been found as a rule that where wax was very close cut it was more stable than a wax with a comparatively large range.

The question of mottling was very largely bound up with the question of air, and his company had really been more concerned with the cure than with the cause. An interesting point was that one might mould a candle under certain conditions and put it away in a warm place and come back in a couple of days and find it all blisters. In fact, he had seen a candle actually with a blow-hole in it, when the temperature had been high enough to soften the wax sufficiently.

With regard to Mr. Gillespie's remark about the curve, his (Mr. Allan's) point had been not so much the curve taking the form it did in bringing the tip to the edge of the flame, as that one could not push the curve back the other way. It would never

burn the other way; it would only burn one way; and the reason why was not clear to him.

The reason why copper stills were used for the distillation of fatty acids was an economic one partly, and as in the stearine process, repeated acid boilings were done, the minute trace of copper, which came off copper condensers, was removed, and it was not in any way harmful to the stearine. Stearine itself very seldom discoloured, except when it was badly made or very old and contained a good deal of oil. On the table in front of him could be seen samples of stearine candles which had been at the Paris Exhibition of 1867. Some were made from palm oil and coconut oil, and the others from palm oil distilled and pressed. They had kept remarkably good colour for all those years.

With regard to the question about beeswax, it was known that the Church had adopted the use of beeswax in the first instance, because it had been the only wax that had been available in very early days; and it had been taken over from the Romans. There was also said to be a reason for it in that the wax had a symbolic meaning from the fact that it was produced by virgin bees. That was said to have some significance in the Church ritual.

A hearty vote of thanks to Mr. Allan was carried with acclamation, and the meeting terminated.

Investigation into the Plastic Properties of Asphaltic Bitumen.*

By Dr. R. N. J. Saal and G. Koens.

Summary.

The viscosities of the most common types of asphaltic bitumen were determined, chiefly below the Ring and Ball temperature, and especially in the neighbourhood of 200 penetration. A capillary viscometer and a viscometer designed by Couette were used for these determinations.

The work done disclosed the fact that the whole matter is exceedingly complicated, for asphaltic bitumen may be :—

1. Plastic; that is to say, its viscosity is not constant, but fluctuates with the shearing stress.
2. Elastic; on the application of a given shearing stress the movement is at first rapid, after which it diminishes until it reaches a constant value. This involves complications in the viscosity determination.
3. Thixotropic; that is to say, in spite of constant shearing stress, the viscosity is not constant, but at first diminishes.

The following conclusions were arrived at :—

(1) In the customary analysis of the asphaltic bitumen the degree of plasticity is expressed in the ductility; inversely, the ductility is a gauge for the plasticity. Roughly, at any rate, it may be said that the flatter the viscosity curve, the more plastic the asphaltic bitumen. Hence blown asphaltic bitumens are more plastic than the steam-refined asphaltic bitumens of the same origin.

(2) There are some asphaltic bitumens which behave altogether like very viscous distillates, and therefore display no plasticity at all. We were able to deduce a formula for the relation between penetration and absolute viscosity of these asphaltic bitumens as follows :—

$$\text{Abs. viscosity} = \frac{5{\cdot}31 \times 10^8}{(\text{pen.})^{1{\cdot}99}} \text{ poises.}$$

This formula applies to penetrations ranging from 5 to 200. Thus the absolute viscosity of a non-plastic asphaltic bitumen increases approximately in inverse ratio to the square of the penetration.

(3) With plastic asphaltic bitumens the difference in viscosity under various shearing stresses may be very substantial. It is highly probable that these differences have a telling effect under working conditions.

(4) Relation between the plasticity and the composition of the asphaltic bitumen.

Maltenes themselves are not plastic; not until asphaltenes are present can there be any plasticity. Nevertheless, the degree of plasticity of an asphaltic bitumen containing a certain percentage of asphaltenes is primarily governed by the nature of the maltenes in that bitumen; the kind of asphaltenes appears to be a matter of secondary importance.

There appear to be three types of maltenes, viz. : (*a*) Those which yield non-plastic or practically non-plastic asphaltic bitumens; (*b*) those which yield asphaltic bitumens with a certain degree of plasticity; (*c*) maltenes

* Paper received June 10th, 1932.

ex typical blown asphaltic bitumens. These yield very plastic asphaltic bitumens.

There are, of course, transitional forms, such as slightly blown asphaltic bitumens.

(5) Comparison between viscosity and penetration.

So far we have only been able to do this at about 200 penetration, when it transpired that, at shearing stresses from 10,000 to 100,000 dynes per sq. cm., asphaltic bitumens of the same penetration have approximately the same viscosity. With other shearing stresses the difference in viscosity naturally depends upon the degree of plasticity.

(6) The viscosity at the Ring and Ball temperature is a somewhat similar case. Thus, for non-plastic asphaltic bitumens we found a viscosity of 7900 poises. This is the viscosity of plastic asphaltic bitumens at that temperature, at a shearing stress of the 100,000 dynes per sq. cm. range.

(7) The influence of the pressure is far greater upon asphaltic bitumens than upon lubricating oils. For instance, under 100 atmospheres, the viscosity of a non-plastic, soft asphaltic bitumen at 35° C. increased to 2½ times its original figure.

Introduction.

The characteristics of the viscosity of asphaltic bitumen are determined by a number of methods for the customary analysis, viz., penetration, Ring and Ball, float test, etc. These determinations can be compared with, for instance, the estimation of the Engler viscosities for the lighter petroleum products, when the viscosity is also measured with an instrument standardised to arbitrarily chosen dimensions.

Besides this Engler viscosity, the absolute viscosity (expressed in poises) is often measured. This is defined as follows :—

Consider two equidistant sheets (surface O) at a distance of 1 cm. from each other with some liquid—say, ordinary lubricating oil—in between and slide these at a constant rate, v, along each other (Fig. 1).

Fig. 1.
SCHEMATIC VISCOMETER.

For this, of course, a certain amount of force—F.O.—is needed. The absolute viscosity, η, is now defined by the equation :—

$$\eta = \frac{F}{v} \text{ (all in C.G.S. units).}$$

Plotted, this relation is curve *a* for ordinary lubricating oils in Fig. 2, a straight line through the origin :—

FIG. 2.

RELATION BETWEEN SHEARING STRESS AND RATE OF SHEAR FOR ORDINARY LUBRICATING OILS (A) AND PLASTIC MATERIALS (B AND C).

For experimentally force F proves to be proportionate to the rate of shear v.

To indicate the order of magnitude of the poise, we may mention the following viscosities (all at room temperature) :—

Kerosine	about	0·02	poise.
Spindle oil	,,	1	,,
Cylinder oil	,,	25	poises.

Now the purpose of this investigation was the determination of the absolute viscosity of asphaltic bitumens, particularly below the Ring and Ball temperature.

It was, however, soon found that the matter is not as simple as for the lighter, distilled petroleum products, since many asphaltic bitumens are plastic.

Plasticity may best be described as follows : Referring to the definition given above of the absolute viscosity, consider mild steel, for instance, instead of lubricating oil. For this a certain amount of force is needed before the metal begins to flow at all. Let this force be F_1 (see Fig. 2), the "yield value." With force F_2, F_1, therefore, flow will take place, say, at the rate of v_2, etc., so that here again we shall have a (generally crooked) line *b* showing the course of the rate of shear with respect to the shearing stress.

Here the viscosity $\left(\frac{F}{v}\right)$ is no longer constant, amounting at F_1 to $\eta = \frac{F_1}{0} = \infty$; at $F_2 : \eta = \frac{F_2}{v_2}$.

There is a third possibility, viz., curve *c*. Here, although the yield value is 0, the viscosity $\eta = \frac{F}{v}$ is not constant because the line is not straight.

Substances with *b* or *c* curves are called plastic. For a more elaborate description, see Bingham[1] and Hatschek.[2]

Now, in our determinations with asphaltic bitumen, the viscosity proved in many instances also to be dependent upon the shearing stress, so that those asphaltic bitumens were plastic. The case of *c* may perhaps represent the behaviour of the softer steam-refined asphaltic bitumens, as under the influence of gravity these flow perfectly straight, so that in any event, with slight F, the viscosity has a finite value, as opposed to many harder asphaltic bitumens (particularly the blown ones) which in the long run do not ostensibly flow out smoothly and therefore more nearly approximate case *b*. By making observations at sufficiently slow rates, it might be possible to discover whether, practically speaking, a yield value occurs here. No choice can, however, be made with certainty between the two cases, because this choice would have to be based on observations made at, mathematically speaking, values of v approaching 0, which is not feasible experimentally.

As a matter of fact, the symptom of "ductility" already points to plasticity. For, if asphaltic bitumens were exceedingly viscous, normal liquids (curve *a*), equal penetration would probably imply equal viscosity and with equal penetration a difference in ductility would be quite inexplicable. For ductility is nothing but a measure for the manner of flowing under certain conditions, and should therefore be explainable by the characteristics of the viscosity. If, on the other hand, asphaltic bitumens are plastic, and that in varying degree, a difference in the manner of flowing of two asphaltic bitumens of the same penetration is far more comprehensible.

We deemed it desirable to institute extensive research into this plasticity of asphaltic bitumens, imposing upon ourselves the condition that the viscosity should be recorded as a function of the actual shearing stress, hence independently of the constants of the apparatus used.

As nothing was known of this matter for asphaltic bitumens,

[1] "Fluidity and Plasticity."
[2] "The Viscosity of Liquids."

our research was primarily tentative. We concentrated mainly upon :—

1. The determination of the plasticity curves (Fig. 2, curve *b* or *c*) for various types of asphaltic bitumen.
2. The search for some connection with the routine determinations, notably penetration, Ring and Ball melting point and ductility.

Disruptive Influences.

In the course of our investigations, the matter proved to be more complicated than was to be inferred from the description given of plasticity, for we were met with the following disruptive influences.

1. *Elasticity.*—It is a known fact that asphaltic bitumens (particularly blown ones) have elastic properties ; they rebound after having been stretched. This has a certain disturbing influence upon the determination of the plasticity. For, in the schematically represented apparatus in Fig. 1, the following process will be found : On applying a constant force, F.O., it transpires that the rate v is not at once constant, but first of all slows down and then, after some time, approaches a constant value v_e (Fig. 3).

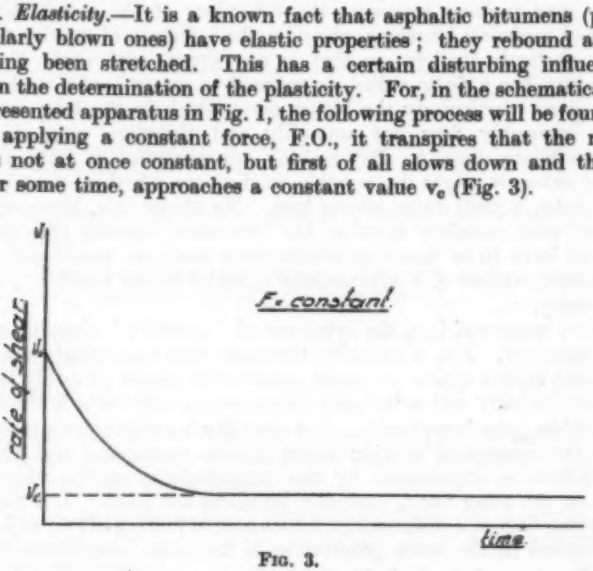


Fig. 3.

RATE OF SHEAR FOR AN ELASTIC ASPHALTIC BITUMEN AS TIME PROGRESSES.

The simplest explanation is as follows :—

If, after v_e has been reached, the force is suddenly reduced, the rate will naturally slow down. If the force is reduced to zero, the asphaltic bitumen will rebound, so that a certain force F' 1·0 will be necessary ($o<F'<F$) to get the asphaltic bitumen to remain stationary. In the simplest case the difference between

$(F-F')$ 1·0 is really the force which causes the rate v_0. At the beginning of the test, when F.o. is started, F′ is still o, so that greater effective force prevails there which causes greater rate, v_0.

2. *Thixotropy.*—This phenomenon follows from, for instance, the test described below :—

Two viscosity determinations were carried out for a blown asphaltic bitumen with capillaries of exactly the same diameter, but of differing length. The pressures with which the asphaltic bitumen was forced through the capillaries were in proportion to the length. It was therefore to be expected that in both cases exactly the same quantities would flow through. This, however, proved not to be the case ; a larger quantity flowed through the longer capillary. As will be elaborated further down, the only possible explanation proved to be that, as a result of its internal movement, the asphaltic bitumen became less viscous. Naturally, this phenomenon, thixotropy (which, as a matter of fact, is one displayed by most plastic substances) greatly complicates the matter. This is extensively dwelt upon in Section VI. and note 5.

On account of these two disruptive influences, elasticity and thixotropy, it is practically impossible to mark down the course which the viscosity of an asphaltic bitumen follows. We were therefore compelled to confine ourselves chiefly to measuring the final values, that is to say, after the asphaltic bitumen has flowed long enough to acquire a constant viscosity.

The effects of the two influences are in contrary directions : through elasticity the viscosity increases during the initial stages of the determination, whereas through thixotropy it decreases.

III.—Equipment.

Two kinds of viscometer were used for this investigation :—

1. *A Capillary Viscometer* (*Fig.* 4).

This is an ordinary model, with which, however, it is possible to apply pressure to the top of the liquid (for asphaltic bitumen pressures up to 10 atmospheres were usually applied). The apparatus was placed in a thermostat maintained at a constant temperature of 0·1° C., and the efflux within a given space of time was measured by cutting off the thread of bitumen under the outflow orifice—always at the same level—before and after the test.

Reproducibility was usually 5 to 10 per cent., all according to the asphaltic bitumen used. These differences must somehow be

connected with the preliminary treatment of the asphaltic bitumen, since duplicate determinations with one and the same charge tallied to within 1 to 2 per cent. Attempts to improve the check values by applying exactly the same preliminary treatment were unsuccessful.

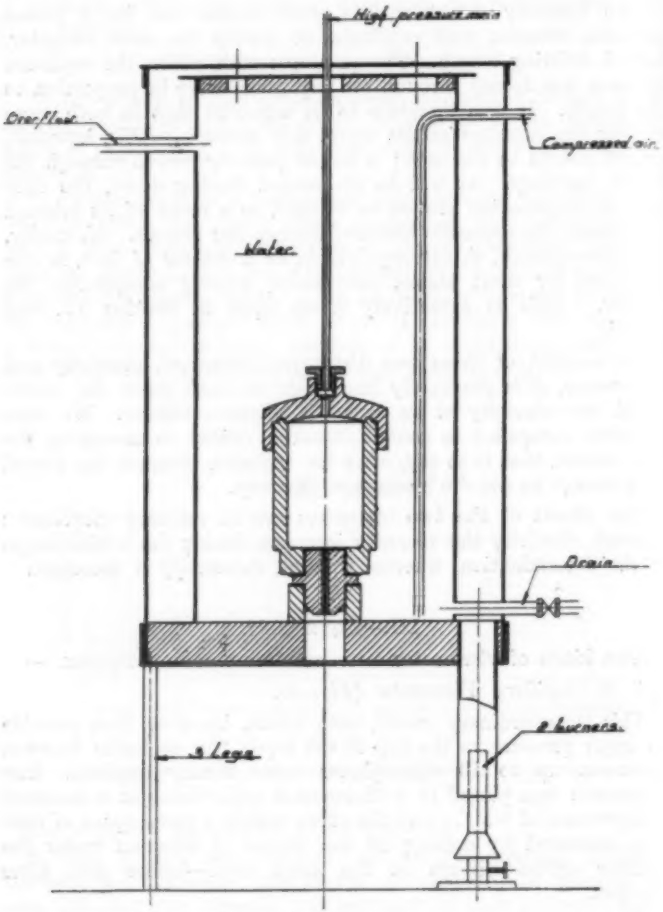


FIG. 4.

CAPILLARY VISCOMETER.

The duration of a determination varied from a few minutes to a few hours, with pressures up to 10 atm.

For liquids whose viscosity follows a normal course (curve *a*, Fig. 1), the viscosity can, of course, be deduced straight away from *Poiseuille's* formula ; but for plastic substances the matter

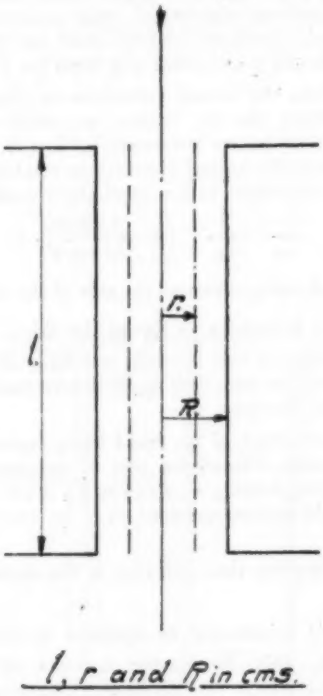


FIG. 5.
CAPILLARY.

becomes more complicated, for it is then a matter of totally different shearing stresses and, therefore, also different viscosities—*e.g.* :—

Force p. π. r^2 acts upon a small cylinder with radius r in the capillary (Fig. 5). The cylinder is carried by its lateral surface

$=2\,\pi.\,r.\,l.$ Hence, the shearing stress per sq. cm. of this surface amounts to :—

$$\frac{p.\,\pi\,.r^2}{2\,\pi\,r.\,l} = \frac{p.\,r}{2\,l}.$$

Therefore this force amounts to o at r=o and $\frac{p.\,R}{2\,l}$ at the l (r=R).

Since, with plastic substances, the viscosity is dependent upon the shearing stress, a series of viscosities exist in the capillary. Therefore, if the viscosity of a plastic substance is determined by means of a capillary viscometer, only an average of all these viscosities is found ; further down we shall call this η_m. So this η_m value is calculated in the usual way from the *Poiseuille* formula.

Now, to calculate the actual viscosities in connection with the shearing stress from the η_m values, we evolved the following formula which, so far as we are aware, makes it for the first time possible to calculate the actual viscosity in relation to the shearing stress from determinations with a capillary viscometer :—

$$\frac{1}{\eta_R} = \frac{1}{\eta_m} \cdot \left\{1 - \frac{d \log \eta_m}{4d \log F_R}\right\}$$

In this, F_R is the shearing stress at the side of the capillary : $\frac{p.\,R}{2\,l}$

η_R is the viscosity belonging to F_R (at the side).

For the deduction of this formula see Addendum, Note 1. By this formula it will be seen that η_m is determined only by F_R, the shearing stress at the side.

Now if determinations of η_m have been made with capillaries of arbitrarily chosen dimensions and at arbitrary pressures, the resultant points on plotting η_m against F_R must lie on one curve, from which the differential quotient—*i.e.*, η_R—is diagrammatically deducible.

Here again, however, the condition is the absence of disruptive influences.

2. *A Concentric Viscometer as designed by Couette* (*Fig.* 6).

Fundamentally, this viscometer consists of two concentric cylinders between which the asphaltic bitumen is placed. The inner cylinder revolves within the outer.

The absolute viscosity can be calculated from the force needed for this (or, rather, the moment) and the resultant angular velocity together with the dimensions of the apparatus. This calculation is far simpler for plastic substances than with the capillary viscometer, and at once shows the relation between viscosity and shearing stress. (See note 2.)

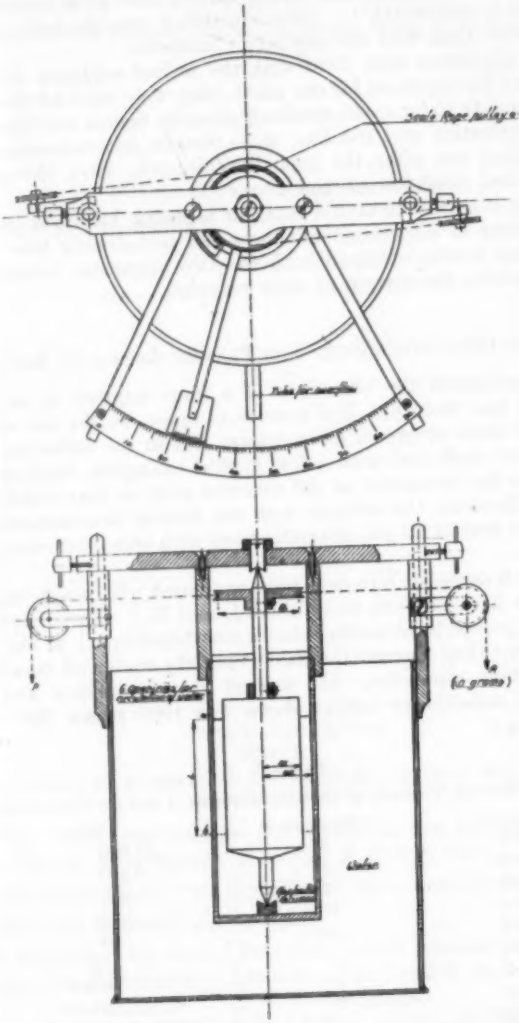


FIG. 6.

A CONCENTRIC VISCOMETER AS DESIGNED BY COUETTE.

The determinations were again carried out at a temperature constant to within 0·1° C. Reproducibility was the same as or a little better than with the capillary viscometer.

Both apparatus were filled with the melted asphaltic bitumens and, after having stood for one night, they were used for the determination. In so far as the resultant viscosity figures were compared with penetration and the like, these routine determinations were also carried out after the asphaltic bitumen, after the pouring process, had stood for one night.

Finally it should be stated that, for asphaltic bitumens in which the presence of disturbing influences had not already been manifested, the results obtained with the two apparatus tallied completely within the margin of error recorded.

IV.—Determinations with Non-Plastic Asphaltic Bitumens.

We determined the viscosities of a large number of asphaltic bitumens (see Note 6). For reasons of space we do not wish to record all these observations *in extenso*, but in the following three sections we shall deal with a few typical examples, alluding with respect to the remainder of the material only to that which may serve to illustrate the relation with the routine determinations.

We now first of all get determinations with non-plastic asphaltic bitumens.

Two such asphaltic bitumens were examined, viz., a soft bitumen (A) and a hard one (B) (see Tables I. and II.). These asphaltic bitumens proved to be neither elastic nor thixotropic ; at the same temperature they showed the same viscosity under all conditions with the two apparatus. The unit of viscosity chosen and also constantly adhered to further down was 1000 poises, the "kilopoise" (k.p.).

Table I.

Relation Between Viscosity of Asphaltic Bitumen A and the Temperature.

Temp. in ° C.	Penetration.*	η in k.p.
5·0	12	42,000
10·0	33	6,800
15·0	71	1,260
20·0	170	240
25·0	250	60
35·0	—	4·9
60·0	—	0·064
75·0	—	0·0110
100·0	—	0·00135
125·0	—	0·000358

* Determined after one night's standing.

TABLE II.

Relation Between Viscosity of Asphaltic Bitumen B and the Temperature.

Temp. in ° C		Penetration.*		η in k.p.
15·0	..	4·5	..	275,000
25·0	..	28·5	..	9,100
30·0	..	63	..	1,680
35·0	..	150	..	347
40·0	..	—	..	89
50·0	..	—	..	8·5

* Determined after one night's standing.

Relation to Penetration.

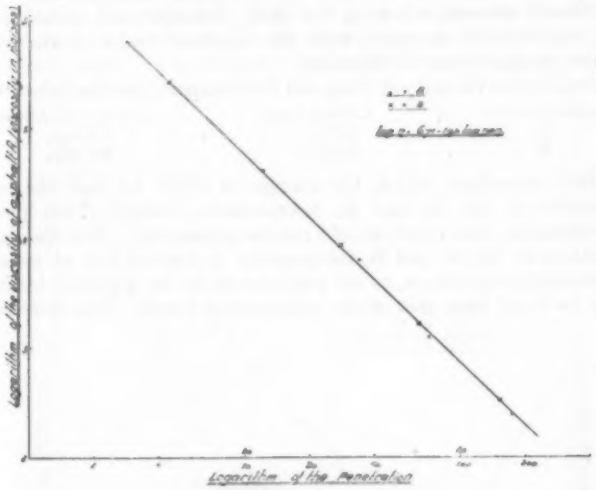


FIG. 7.

RELATION BETWEEN PENETRATION AND ABSOLUTE VISCOSITY FOR ASPHALTIC BITUMEN A AND B.

In Fig. 7 the penetrations and viscosities are plotted against each other (as logarithms); from this it is seen that :—

1. The points for the two asphaltic bitumens conjoin on one line.
2. This line is practically straight.

It is therefore easy to represent the relation between penetration and absolute viscosity by a formula. This formula is :—

$$\eta = \frac{5{,}13 \cdot 10^6}{(\text{penetration})^{1 \cdot 53}} \text{ k.p.} = \frac{5{,}13 \cdot 10^9}{(\text{penetration})^{1 \cdot 53}} \text{ poises.}$$

Hence this formula applies to penetrations between 5 and 200, and can be used as conversion formula for non-plastic, very viscous

substances, always provided the adhesion to the penetrometer needle be always the same. A theoretical deduction of this formula is given in Note 3.

Thus the determination of the penetration may be conceived as a determination of the viscosity. If, however, a plastic asphaltic bitumen is under examination, this determination can at the most stand in relation to a kind of average viscosity. It is generally the custom to compare two asphaltic bitumens of the same penetration; the above formula now makes it possible to calculate from the penetration of the plastic asphaltic bitumen the viscosity of a non-plastic asphaltic bitumen of the same penetration. This calculated viscosity (which we shall henceforward indicate by η_{pen}) can then be compared with the observed series of viscosities of the plastic asphaltic bitumen.

Further, the viscosity at Ring and Ball temperature was deduced:—

Asphaltic bitumen.		R. and B. temp.		η at that temperature.
A	..	33° C.	..	7·6 k.p.
B	..	50·2° C.	..	8·1 k.p.

Here, therefore, within the margin of error, we find the same viscosity at the R. and B. temperature, notably 7900 poises. Nevertheless, this result should not be generalised. For the determination of the R. and B. temperature is carried out at steadily increasing temperature, so the temperature in the asphaltic bitumen may be lower than that of the surrounding water. Therefore, with

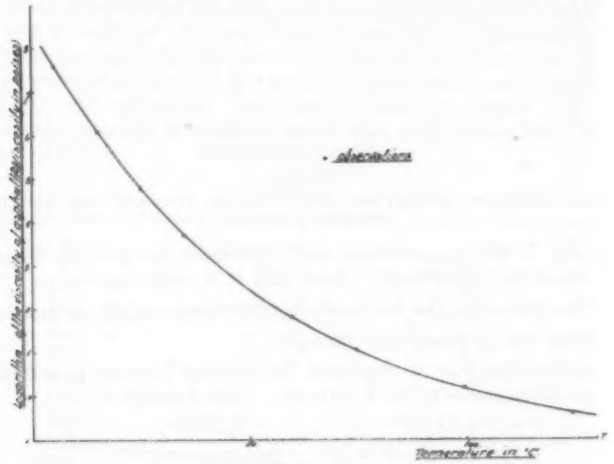


FIG. 8.

RELATION BETWEEN VISCOSITY AND TEMPERATURE FOR ASPHALTIC BITUMEN A.

entirely different coefficients of temperature there may be differences in the viscosity at the R. and B. temperature. Moreover, the conductivity of heat may also be different, while also, of course, the bitumen begins to fall through at far lower temperature.

The course of the viscosity of the asphaltic bitumens is steadliy continuous from 5 to 125° C. (Fig. 8).

Mixtures of Asphaltic Bitumen and Light Oils.—Determinations were also carried out with mixtures of asphaltic bitumen A and light oils, viz., with :—

1. Liquid paraffin E° 20 = 14·2.
2. Spindle oil Edeleanu extract.

In Tables III. and IV., and in Fig. 9, we give the results at 15° C. At this temperature the viscosities of the two oils are practically alike. The mixtures displayed no plasticity.

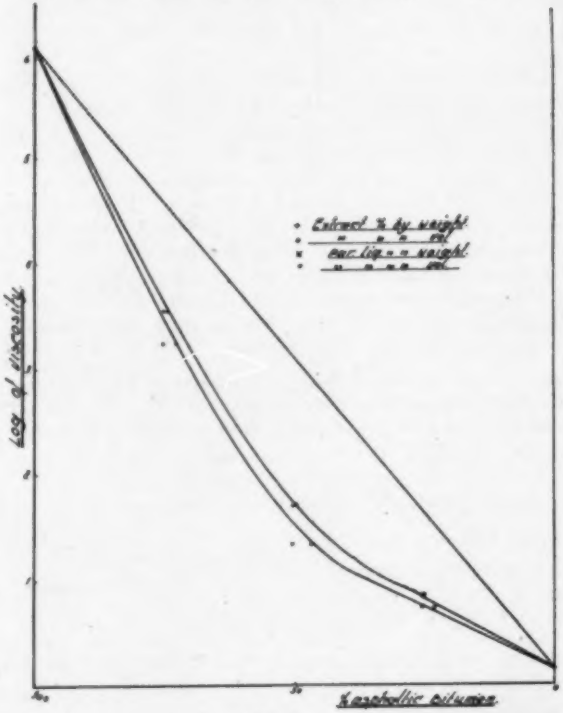


FIG. 9.

VISCOSITY OF MIXTURES OF ASPHALTIC BITUMEN A AND LIGHT OILS 15°.

TABLE III.

Viscosity of Mixtures of Asphaltic Bitumen A and Liquid Paraffin.

Per cent. by wt. asph. bit.	100	75	50	25	0
Per cent. by vol. asph. bit.	100	72·4	46·4	22·4	0
Per cent. mol. per cent. asph. bit.	100	69·8	43·7	20·3	0
η (poises)	$1.26.10^6$	$1.85.10^3$	39.6	5.30	1.31

(Average mol. weight asphaltic bitumen, 460; of liquid paraffin, 355.)

TABLE IV.

Viscosity of Mixtures of Asphaltic Bitumen A and Edeleanu Extract.

Per cent. by wt. asph. bit.	100	75	50	25	0
Per cent. by vol. asph. bit.	100	74·3	49·5	24·5	0
Per cent. mol. per cent. asph. bit.	100	62·5	35·7	15·6	0
η (poises)	$1.26.10^6$	$3.7.10^3$	90.1	7.07	1.33

(Average molecular weight of Edeleanu extract, 255.)

Notwithstanding the fact that the viscosities of the two oils are the same, in none of the three ways could we get the curves for liquid paraffin and Edeleanu extract to tally. We found the same at whatever other temperature we determined these curves. The curves for per cent. by volume approach the nearest to each other. Although this result need not cause the least surprise, we thought it as well once more to draw special attention to it, as this may be of importance in connection with the fluxing of asphaltic bitumens, for in that, too, similar differences are sometimes found. One might sometimes feel inclined to ascribe this to the asphaltenes, but it follows from Fig. 9 that the maltenes may already produce differences in this respect.

Finally, a test was made of the influence of the pressure upon the viscosity. These tests were made with the help of a pressure vessel, which could be fitted to the bottom of the capillary viscometer. The pressures at the bottom and the top of the capillary were determined with precision manometers.

It transpired that the influence of pressure was very great.

Asphaltic Bitumen A.

Viscosity under an average of 5 atm. : 5·0 k.p.
Viscosity under an average of 105 atm. : 12·2 k.p. } ratio 2·45.

(Temperature 35° C.)

According to Kieszkalt, among others, the following formula applies to mineral and fatty oils :—

$$\eta_p = \eta_o \cdot 10^{\frac{p}{p_e}}$$

In this :

η_p is the viscosity under pressure p.

η_o is the viscosity under pressure o.

p_o is a constant.

For lubricating oils p_o is of the order of magnitude of 1000 atm.

If we accept this formula as correct, then it follows from our observations that p_o=250 atm. for asphaltic bitumen A ; that is to say that under 250 atm. the viscosity is already 10 times greater than at ordinary pressure.

V.—Determinations With a Normal Steam-Refined Asphaltic Bitumen.

Our next example is asphaltic bitumen C, a normal, steam-refined asphaltic bitumen, penetration 178 at 25° C. With the Couette viscometer we obtained the following :—

Table V.

Course of the Viscosity of Asphaltic Bitumen C. at 25° C.

F (in dynes).		η (in k.p.).		$\frac{dv}{dr}$
610	..	258	..	0·0024
1,220	..	250	..	0·0049
2,440	..	246	..	0·0100
12,200	..	218	..	0·056

In this F = shearing stress in C.G.S. units.

$\frac{dv}{dr}$ = rate of shear in C.G.S. units.

To afford an impression of the angular velocities observed we will mention that the number of seconds per 1 degree of rotation at a F of 1220 dynes is found by dividing η in k.p. by about 15.

It follows from the table that this asphaltic bitumen is plastic. Determinations were also made with the capillary viscometer. We should like to make the general observation that in determinations with the capillary viscometer the weighed, snipped thread of asphaltic bitumen was a good length (in the region of about ½ m.). The following figures were found :—

Table VI.

Course of Viscosity of Asphaltic Bitumen C. at 25° C.

R (cm.)	$p\left(\frac{kgs}{cm^2}\right)$	η_m (k.p.)	$F_R=\frac{p.R}{2\,l}$ (dynes)	η_R (k.p.)
0·147	5·60	202	$84\cdot10^3$	199
0·147	9·90	193	$146\cdot10^3$	190
0·249	5·60	195	$140\cdot10^3$	192
0·249	9·90	188	$247\cdot10^3$	185

1 cap = 4·9 cms.

η_m and F_R, when plotted against each other, give points which come on the same curve; presumably, therefore, there are no disturbing elements.

(The correction required to correct η_m to η_R—see Note 1—proved to be small (1½ per cent.) and is therefore really superfluous here. Without the calculation, however, the order of magnitude of the correction would have remained entirely unknown to us.)

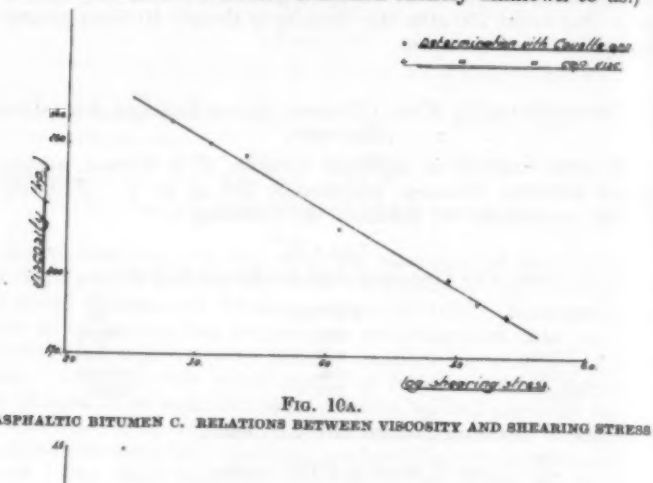


FIG. 10A.

ASPHALTIC BITUMEN C. RELATIONS BETWEEN VISCOSITY AND SHEARING STRESS

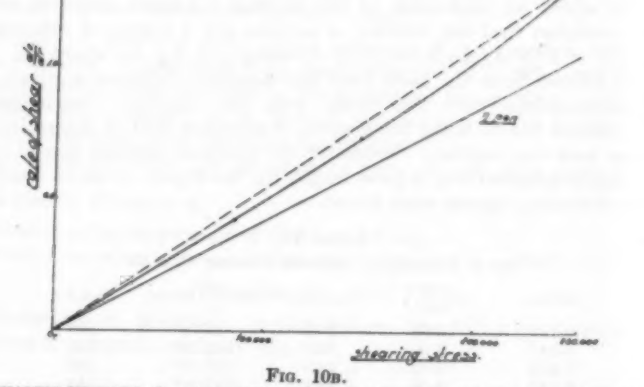


FIG. 10B.

ASPHALTIC BITUMEN C. RELATION BETWEEN SHEARING STRESS AND RATE OF SHEAR.

If the two results are plotted, they come properly on one curve (see Fig. 10a, log F against η) ; in Fig. 10b the ordinary plasticity curve (as per Fig. 2) is plotted. The deviation from the curve *a* in Fig. 2 is, therefore, not strikingly great.

The penetration of the asphaltic bitumen at 25° C. amounted to 178 ; hence $\eta_{pen.}$ amounts to 237 k.p. (see Fig. 7). With C, this viscosity is within the series of measured viscosities. Thus with plastic substances, the determination of the penetration seems to indicate a kind of average viscosity (also see Section VII.).

Determinations With a Very Plastic Asphaltic Bitumen.

As we saw, asphaltic bitumens A and B were without plasticity and without disturbing elements ; asphaltic bitumen C was found to be plastic, but no disruptive influences were encountered ; in asphaltic bitumen D, a hard, blown bitumen (pen. 34 at 25° C.), which we extensively examined at 75° C., we found not only plasticity, but also disturbing elements.

With the Couette apparatus the whole is still surveyable, because measuring can be deferred until the final value is reached after sufficiently lengthy revolving. When power is started, the cylinder revolves at first rapidly, then more slowly, and in the course of about a quarter of a minute reaches a constant speed. Clearly, therefore, elasticity is present ; it is difficult to indicate thixotropy on account of the short space of time. The following was found :—

Table VII.

Course of Viscosity of Asphaltic Bitumen D at 75° C.

F.	η (k.p.).	$\frac{dv}{dr}$
390	204	0·0019
710	189	0·0038
1280	169	0·0076
2120	140	0·0151
4360	114	0·038
7100	94	0·076
11300	75	0·151

Note 4 shows that there is no slip along the wall here.

With the capillary viscometer, on the other hand, the behaviour is very complex ; the following η_m values were determined :—

Table VIII.

Course of Viscosity of Asphaltic Bitumen D at 75° C.

Pressure (kgs/sq. cm.).		4·69	5·40	5·85	7·95	9·90
R = ...	η_m(k.p.)	35·1	29·0	27·4	18·8	14·4
0·118 cm. ..	F_R	$55 \cdot 10^3$	$64 \cdot 10^3$	$68 \cdot 10^3$	$94 \cdot 10^3$	$117 \cdot 10^3$
R = ...	η_m(k.p.)	19·8	16·8	15·0	10·3	8·2
0·147 cm. ..	F_R	$69 \cdot 10^3$	$79 \cdot 10^3$	$86 \cdot 10^3$	$117 \cdot 10^3$	$146 \cdot 10^3$
R = ...	η_m(k.p.)	11·4	10·0	9·0	6·2	5·3
0·249 cm. ..	F_R	$117 \cdot 10^3$	$134 \cdot 10^3$	$145 \cdot 10^3$	$198 \cdot 10^3$	$247 \cdot 10^3$
R = ...	η_m(k.p.)	9·2	7·2	6·7	4·4	3·2
0·388 cm. ..	F_R	$182 \cdot 10^3$	$209 \cdot 10^3$	$227 \cdot 10^3$	$308 \cdot 10^3$	$384 \cdot 10^3$

l_{cap} = 4·9 cms.

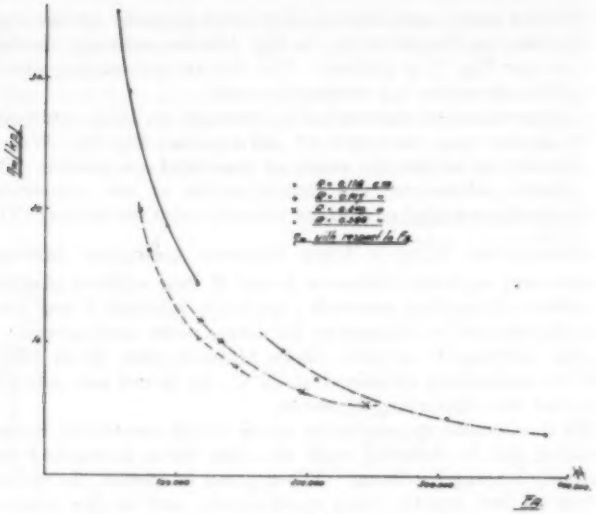


FIG. 11.

RELATION BETWEEN η_m AND F_R FOR ASPHALTIC BITUMEN D.

If these η_m values are plotted against F_R (Fig. 11), the points no longer conjoin on one curve, which implies the presence of disturbing influences. It can then be shown that this must chiefly be thixotropy (see Note 5). This was proved particularly by the experiment described under "Thixotropy" in Section II. of this report, that experiment being further figured out in Note 5. For instance, to get equal quantities of asphaltic bitumen to flow through a capillary of 0·107 cm. radius, the following was required :—

With 4·9 cms. length .. .. 5·0 atm.

With 25 cms. length .. .. $\frac{25}{4 \cdot 9} \cdot 3 \cdot 7$ atm.

Consequently, a further accurate calculation cannot be made regarding these observations; their importance, however, lies in the great difference between them and the observations made with the Couette viscometer, which is a clear indication of the plastic character of the asphaltic bitumen.

The thixotropy can, of course, for once be left out of consideration and, say, from a series at equal $\frac{p}{l}$ calculate the η_R. From observations recorded in Table VIII. one then finds the following :—

TABLE IX.

Course of Viscosity of Asphaltic Bitumen D at 75° C.

p = 4·60 kos/sq. cm.			p = 9·90 kos/sq. cm.		
η_R (k.p.)	F_R	$\left(\frac{dv}{dr}\right)_R$	η_R (k.p.)	F_R	$\left(\frac{dv}{dr}\right)_R$
17·1	$55 \cdot 10^3$	3·2	6·9	$117 \cdot 10^3$	17·0
13·9	$69 \cdot 10^3$	5·0	5·6	$146 \cdot 10^3$	26·1
9·8	$117 \cdot 10^3$	11·9	3·85	$247 \cdot 10^3$	64
8·2	$182 \cdot 10^3$	22·2	2·8	$384 \cdot 10^3$	137

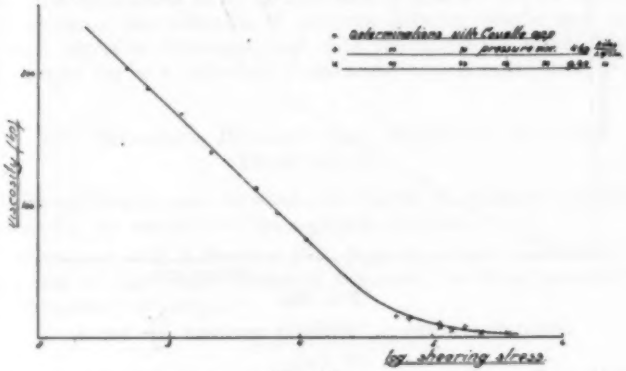


FIG. 12A.

ASPHALTIC BITUMEN D. RELATION BETWEEN VISCOSITY AND SHEARING STRESS.

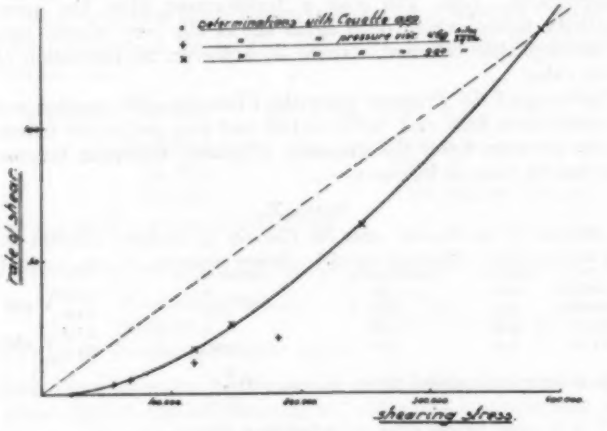


FIG. 12B.

ASPHALTIC BITUMEN D. RELATION BETWEEN RATE OF SHEAR AND SHEARING STRESS.

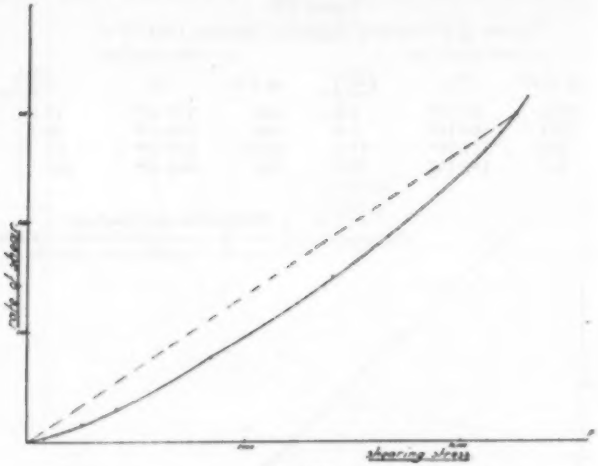


FIG. 12c.

ASPHALTIC BITUMEN D. RELATION BETWEEN $\frac{dv}{dr}$ AND F.

If these (inexact) figures are plotted (Fig. 12A), they will be found to come fairly satisfactorily in the extension of the series of Couette observations. Figs. 12b and c furthermore give the normal plasticity figure, which once again shows the very plastic nature of the asphaltic bitumen. There is, however, no indication of a yield value.

Influence of the Pressure upon the Viscosity.—We carried out a determination here and there to feel our way as to the influence of the pressure upon the viscosity of plastic asphaltic bitumens. The results were as follows :—

TABLE X.

Influence of the Pressure upon the Viscosity of Asphaltic Bitumen D.

R cap. (cm.).	L cap. (cm.).	Pressure at top cap. (kgs/sq. cm.).	Pressure bottom cap. (kos/sq. cm.).	η_m.	p_o.
0·0614	4·9	20	0	10·0	495
0·0614	4·9	100	80	14·6	
0·118	4·9	10	0	14·2	435
0·118	4·9	110	100	24·0	

p_o is again calculated from $\eta p = \eta_o \cdot 10^{\frac{p}{p_o}}$

If it is now taken into consideration that :—

1. The influence of the pressure upon lubricating oils diminishes at higher temperature, that is to say that p_o increases ;

2. The influence of the pressure upon heavier petroleum products, compared at the same temperature, is usually greater than upon the lighter kind ;
3. The maltenes of asphaltic bitumen D at room temperature were less viscous than those of asphaltic bitumen A ;

then it follows from comparison with the value $p_c = 250$ atm. for asphaltic bitumen A at 35° C. that there is in any event no essential difference in the influence of pressure between plastic and non-plastic asphaltic bitumens, but that the difference must chiefly be sought for in a difference in maltenes and temperature.

VII.—Relation Between the Plasticity and the Penetration.

The question now is : to what extent is the penetration a sufficient gauge for the viscosity of the asphaltic bitumen ?

As we saw with A (Section IV.), there is a direct connection, in the case of non-plastic asphaltic bitumens, between penetration and viscosity, notably :—

$$\eta = \frac{5 \cdot 13 \cdot 10^9}{(\text{pen.})^{1 \cdot 93}} \text{ poises.}$$

It furthermore transpired with C (Section V.) that the $\eta_{pen.}$ comes in the range of measured viscosities, so that it would seem that the penetration there measures a species of average viscosity.

This also proved to be the case with more plastic asphaltic bitumens, *e.g.* :—

1. Asphaltic bitumen D 60° C. $_{pen.}=180$ $\eta_{pen.}= 232$.

F=525 dynes	η=3300 k.p.
21,000	1030
200,000	<98 (about 70 k.p.)

$\eta = \eta_{pen.}$ at F=about 75,000 dynes.

2. Asphaltic bitumen E (blown) 25° C. pen. 190 $\eta_{pen.}=207$.

F=1100 dynes	η=850 k.p.
200,000	<110 (ab ·75 k.p.)

$\eta = \eta_{pen.}$ at F=about 100,000 dynes.

With these asphaltic bitumens (all of which have a penetration of about 200 at the temperature at which the measurement was made), $\eta_{pen.}$ lies in the area of the measured viscosities.

In Fig. 13 we have plotted log $\frac{\eta_{F=12,000}}{\eta_{pen.}}$ against the penetration

(*i.e.*, observations at different temperatures).* It follows from the fact that, with greater shearing stress the curves in the figure come lower (the curve for $\eta_{F=200,000}$ is lower than the curve for $\eta_{F=12,000}$), that, with lower penetrations, the shearing stress at which η becomes equal to $\eta_{pen.}$ is greater. Up to a certain

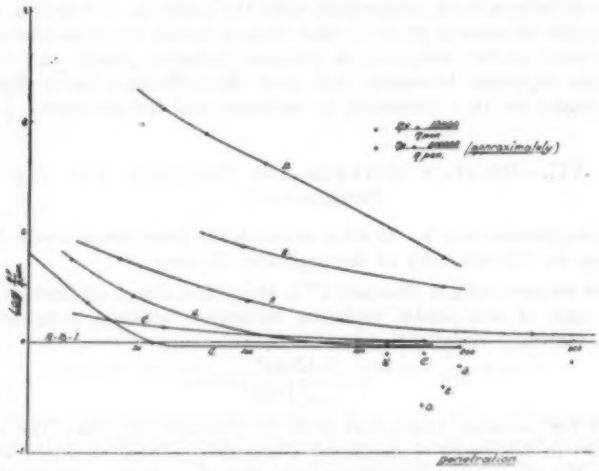


FIG. 13.

COURSE OF $\frac{\eta_F}{\eta_{pen}}$ WITH THE PENETRATION OF ASPHALTIC BITUMENS.

point this is feasible : the needle penetrates less deeply, is therefore on an average carried by a smaller surface, so that the average shearing stress may be greater. Possibly, too, the asphaltic bitumens become more plastic at lower temperature. Our observations in the case of asphaltic bitumen D certainly point to this, but, owing to the thixotropy, are not sufficiently reliable. For we found :—

	60° C.	75° C.	85° C.
η at F = 12,000	1400 ..	70 ..	28
η at F = 200,000	65 ..	4·5 ..	2·0
Ratio ..	22 ..	16 ..	14

Then, of course, it should not be forgotten that our viscosity determinations are final values, that is to say, are not disturbed by

* At lower penetration, all asphaltic bitumens show a greater ratio :— $\frac{\eta_{F=12,000}}{\eta_{pen.}}$

elasticity or thixotropy. In the determination of the penetration, this is far more complicated.

The conclusion is, therefore, that :—

Soft asphaltic bitumens of equal penetration have as a rule equal viscosity at a shearing stress lying between about 10,000 to 100,000 dynes per sq. cm. At other shearing stresses the viscosity then further depends upon the degree of plasticity.

Approximately, therefore, the penetration yields a sufficient gauge for the viscosity, but for finer differences, the plasticity must at once be taken into account.

For harder asphaltic bitumens we have not yet been able to establish this definitely. The subject would seem to us to be too complicated to permit of generalising and applying the above result to harder asphaltic bitumens, but the following may be said in this regard :—

If the relation of the viscosities at two shearing stresses (say, F = 12,000 and F = 200,000, Fig. 14), the order of magnitude does not usually alter with the temperature in the field we have

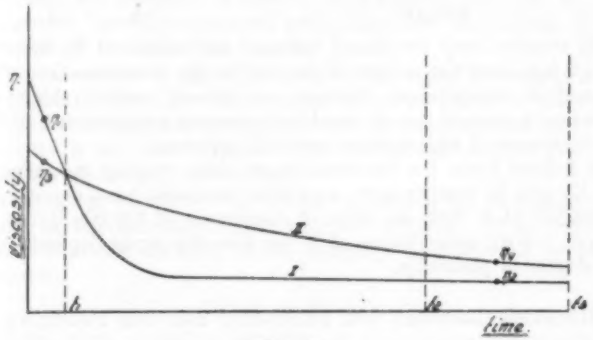


FIG. 14.

VISCOSITY FOR A THIXOTROPIC ASPHALTIC BITUMEN AS TIME PROGRESSES

explored. For this see the values just given for asphaltic bitumen D. For the time being, this relation (measured at a penetration of about 200) can, therefore, be regarded as a gauge for the plasticity. It then strikes one that the most plastic substances at the same time show the largest figures for $\frac{\eta F=12,000}{\eta pen.}$ so that, inversely, from this relation a rough estimate can be had of the degree of plasticity.

VIII.—Relation Between the Plasticity and the Ring and Ball Temperature.

The viscosity of the asphaltic bitumens A and B found at the Ring and Ball temperature was 7·9 k.p. The viscosities of a few other asphaltic bitumens found at their R. and B. temperature were :—

1. Asphaltic bitumen D. R. and B. temperature 85° C.

F (dynes).	η (k.p.)
525	58
200,000	Abt. 2

So here, too, the value found with A and B is within the area. At η=7·9 k.p., F amounts to about 50,000 dynes.

2. Asphaltic bitumen F. R. and B. temperature 39½° C.

F (dynes).	η (k.p.)
600	13·5
2400	12·4

3. Asphaltic bitumen G. R. and B. temperature 41·5° C.

F (dynes).	η (k.p.)
600	11·5
140,000	7·9

No relation may be placed between the values of F, in which η=7·9 k.p., and the weight of the ball in the determination of the R. and B. temperature, because, as already stated, this determination is carried out at steadily increasing temperature, so that the coefficient of temperature may still influence.

It follows from the determinations that, roughly speaking, at the R. and B. temperature, asphaltic bitumens have about equal viscosities at F with an order of magnitude of 100,000 dynes per sq. cm. With other values of F the viscosity again depends upon the degree of plasticity.

Relation Between the Plasticity and the Ductility.

In the following table we give the ductilities of some of the asphaltic bitumens we examined :—

Table XI.

Ductility of some of the Asphaltic Bitumens examined.

Asphaltic Bitumen.	0° C.	15° C.	25° C.	40° C.	50° C.
A	0	..100	—	—	—
C	35	..100	—	—	—
D	0	2·5	3	5	6
E	6	44	82	>100	—
F	36·5	..100	—	—	—
G	50	..100	—	—	—

On comparison with Fig. 13 it at once strikes one that asphaltic bitumens D and E, which have a flat ductility curve, are at the same time the most plastic (the degree of plasticity regarded as the difference in viscosity, that is to say, in Fig. 13, the distance between the points for F=12,000 and F=200,000 at a penetration of about 200). With higher penetrations, the following explanation may be offered :—

In the ductility determination, the shearing stress is greatest at the smallest section; with a plastic asphaltic bitumen, therefore, the viscosity is lower there than in the wider parts. Consequently, with a plastic bitumen, the stretch at the smallest section (as compared with the larger sections) will be more intense than in the case of non-plastic asphaltic bitumen; the wide parts yield less material, as it were, for the forming thread than with non-plastic bitumens; as a result the thread will be thinner and, therefore, will sooner break.

It further transpired from the ductility determinations that with low penetrations the ductility of a very plastic asphaltic bitumen may be still greater than o, while less plastic bitumens then at once break through. In this the elasticity (which with blown bitumens, for instance, is always considerable) and the, at first changeable, viscosity connected with it (see Sect. II. and Fig. 3) will undoubtedly be influential factors.

The differences in ductility of the other asphaltic bitumens are too slight to reveal an interrelation with the degree of plasticity. All the ductilities increase rapidly as the temperature increases; the plasticity is far less in degree than in the case of the first two asphaltic bitumens in the table.

Here again, moreover, the whole question is too complicated to permit of the indication of an accurate relation. It is, however, certain that the ductility curve primarily indicates the degree of plasticity.

Cause of Plasticity.

Seeing that asphaltic bitumen A (containing practically no asphaltenes) is not plastic, the obvious course is to seek for the cause of plasticity in the asphaltenes. Indeed, we were unable to show any plasticity in the case of the maltenes—*e.g.* :—

Maltenes ex asphaltic bitumen D :—

Asphaltene content 0·45 per cent.
60° C. Vogel-Ossag viscometer.

Efflux under own weight	= 3·36 poises	Ratio shearing stresses about 1 to 5.
Efflux under 60 cms. water pressure	= 3·30 ,,	

In the asphaltic bitumen itself, the ratio of 1 to 5 shearing stress would result in a ratio of the viscosities of about 4 to 1; here, however, the difference is on the border-line of the error of observation. Nor did maltenes of normal steam-refined bitumens give any noticeable plasticity.

There are several indications to show that it is plausible that the nature of the asphaltenes is of only secondary importance, and that the plastic properties of asphaltic bitumen are primarily governed by the nature of the maltenes. Thus we found that a mixture of maltenes ex asphaltic bitumen D+asphaltenes ex the steam-refined asphaltic bitumen H had exactly the same very plastic properties of asphaltic bitumen D, whereas, conversely, maltenes ex asphaltic bitumen H+asphaltenes ex D produced a typical steam-refined asphaltic bitumen. Within certain limits (24–32 per cent.), the percentage of asphaltenes in the mixtures here proved to be of subsidiary importance to the degree of plasticity as compared with the influence of the maltenes. Hence the plastic properties of an asphaltic bitumen are primarily governed by its maltenes; the kind of asphaltenes appears to be of subordinate importance in this respect. Roughly, it may be said that the maltenes indicate the order of magnitude of the plasticity with an equal percentage of asphaltenes, while the percentage of asphaltenes will further be about proportionate to the degree of plasticity.

If one now considers the measured plasticities, one first of all finds a distinction between steam-refined and blown asphaltic bitumens of the same origin, and secondly between I (non-plastic) and, say, C (plastic), which two asphaltic bitumens have about the same percentage of asphaltenes. For the time being, therefore, we should feel inclined to distinguish between three types of maltenes, viz.:—

(*a*) Those which yield non-plastic or practically non-plastic asphaltic bitumens (I).

(*b*) Those which yield asphaltic bitumens with a certain degree of plasticity, to which class C, G and H belong.

(*c*) Typically blown asphaltic bitumens (very plastic).

There are, of course, transitional forms, such as slightly blown asphaltic bitumens.

In the foregoing we have described our investigations into the plastic properties of asphaltic bitumens at penetrations of about 200. This, of course, by no means closes the subject. More knowledge is desirable, particularly with regard to the lower penetrations. Investigations which we carried out into this matter showed a greatly magnified influence of disturbing elements upon the determinations. So far as elasticity is concerned, this difficulty

will no doubt be surmountable, particularly if this property has been more systematically examined at higher penetrations; the thixotropy, however, will probably make it impossible to submit a rounded-off presentation of the whole problem for hard asphaltic bitumens in the same way as above for bitumens of about 200 penetration.

Addendum.

Note 1.—This formula is deduced from the two following principles :—

(1) The shearing stress at the side F_R amounts to $\frac{p.R}{2.l}$.

If for two different capillaries 1 and 2, this is equal to:

$$\frac{p_1.R_1}{2.l_1}=\frac{p_2.R_2}{2.l_2}$$

then the viscosity also is equal at the side in both cases (so long, at least, as no disruptive influences are present). The same applies, for instance, also to the half radii, etc., so it is possible that also the η_m values are the same in both cases. This, indeed, proves to be the case.

The definition of viscosity is :—

$$\eta=\frac{F}{v}$$

in which :—

F is the shearing stress, hence on radius r (Fig. 5) $\frac{p.r}{2.l}=F_r$.

v is the difference in rate per 1 cm. distance; more generally written thus :—

$$\frac{dv}{dx},$$

in which dv indicates the difference in speed at a distance of dx.

For a layer in the capillary between r and r+dr the formula becomes :—

$$\eta_r=\frac{\frac{p.r}{2.l}}{-\frac{dv}{dr}}$$

Further, suppose that $r=a.R$ and, since η_r is merely a function of F_r (presuming disruptive influences to be absent),

$$\eta_r=f_1\left(\frac{p.r}{2.l}\right)=f_1\left(a.\frac{p.R}{2.l}\right)=f_1(a.F_R),$$

then the formula becomes

$$f_1(\alpha.F_R)=\frac{\alpha.F_R}{\frac{1}{R}\cdot\frac{dv}{d\alpha}} \text{ or}$$

$$\frac{dv}{d\alpha}=-\frac{\alpha.F_R.R}{f_1(\alpha.F_R)}.$$

Integrated, this becomes (F_R and R are constant) :—

$$v_\alpha=R.f_2(\alpha, F_R)+C_{int.}$$

(in which, therefore, f_2 is a certain function of α and F_R.

With $\alpha=1$ (at the side) $v=0$, thus

$$v_\alpha=R\,[f_2(\alpha, F_R)-f_2(1, F_R)]$$

This formula, therefore, indicates the rate of flow at distance $\alpha.R$ from the axle of the capillary.

The effluxed volume per second, V, is then

$$V=\int_{r=0}^{r=R}2.\pi.r.dr.v_r$$

$$=2.\pi.R^3\int_{\alpha=0}^{\alpha=1}\alpha.d\alpha.R\,[f_2(\alpha, F_R)-f_2(1, F_R)]$$

$$=2.\pi.R^3\left[\begin{matrix}\alpha=1\\ f_3(\alpha, F_R)\\ \alpha=0\end{matrix}\right]$$

$$=2.\pi.R^3.f_4(F_R).$$

V is therefore a function of R and F_R.

Now as η_m is calculated with the help of *Poiseuille's* formula :—

$$V=\frac{\pi.p.R^4}{8.\eta.l}, \text{ hence } \eta_m=\frac{\pi.p.R^4}{8.l.V},$$

one can also write :—

$$\eta_m=\frac{\pi.p.R^4}{8.l}\cdot\frac{1}{2.\pi.R^3.f_4(F_R)}$$

$$=\frac{1}{8}\cdot\frac{p.R}{2.l}\cdot\frac{1}{f_4(F_R)}$$

$$=\frac{1}{8}\cdot F_R\cdot\frac{1}{f_4(F_R)}$$

In other words, η_m is merely a function of F_R and otherwise does not depend upon the particular dimensions of capillary, etc.

Reiner[3] arrived, along a somewhat different path, at a similar result.

[3] J. Rheo., 1930, 1, 250.

In the absence of disruptive influences, therefore, equal η_m values must be found with

$$\frac{p_1.R_1}{2.l_1}=\frac{p_2.R_2}{2.l_2}$$

If, therefore, one plots the η_m values, obtained with several capillaries, each with several pressures, against the accompanying

$$F_R=\frac{p.R}{2.l}$$

then all points must lie on one curve. If they do not do this, disturbing influences (elasticity or thixotropy) must have played a part.

(2) Now, to deduce the actual magnitude of the viscosity with respect to the shearing stress from the η_m values, we compare, with equal drop in pressure $\frac{p}{l}$, two capillaries with radii R and R+dR.

The shearing stresses between r=o and r=R are, with equal r, the same in both cases, viz.,

$$\frac{p.r.}{2.l}$$

The only difference between the two cases is that with the radius R+dR the entire piece between r=o and r=R has acquired a certain additional speed. Regarded as a whole, this piece flows "internally" in exactly the same way as with the capillary with radius R. This increase in the speed amounts to

$$dv_R=\frac{F_R}{\eta_R}\cdot dR$$

in which

η_R=the viscosity at the side (belonging to F_R).

For, the definition of viscosity is :—

$$\frac{\text{speed at distance a from the side}}{a}=\frac{\text{shearing stress}}{\text{viscosity}}$$

The augmentation of the volume flowing through per second, dV, then amounts to :—

$$dV=\pi\cdot R^2\cdot dv_R=\pi\cdot R^2\cdot\frac{F_R}{\eta_R}\cdot dR$$

This formula only holds good with equal $\frac{p}{l}$, so

$$\left(\frac{dV}{dR}\right)_{\frac{p}{l}}=\pi R^2\times\frac{F_R}{\eta_R}$$

Further, η_m is deduced from *Poiseuille's* formula :—

$$V=\frac{\pi.p.R^4}{8.\eta_m.l}$$

This, differentiating at constant $\frac{p}{l}$, yields

$$\left(\frac{\delta V}{\delta R}\right)_{\frac{p}{l}}=\frac{\pi}{8}\cdot\frac{P}{l}\left\{\frac{4R^3}{\eta_m}-\frac{R^4}{\eta_m^2}\left(\frac{\delta\eta_m}{\delta R}\right)_{\frac{p}{l}}\right\}$$

$\left(\frac{\delta V}{\delta R}\right)_{\frac{p}{l}}$ eliminated from the two formulas yields :—

$$\frac{1}{\eta_R}=\frac{1}{\eta_m}\left\{1-\frac{R}{4\eta_m}\left(\frac{\delta\eta_m}{\delta R}\right)_{\frac{p}{l}}\right\}$$

$$=\frac{1}{\eta_m}\left\{1-\left(\frac{\delta\ln\eta_m}{\delta\ln R^4}\right)_{\frac{p}{l}}\right\}$$

Instead of $\frac{R}{4\eta_m}\left(\frac{\delta\eta_m}{\delta R}\right)_{\frac{p}{l}}$ one may write :—

$$\frac{p.R}{2.l}\cdot\frac{1}{4\eta_m}\cdot\left(\frac{\delta\eta_m}{\delta\frac{p.R}{2.l}}\right)_{\frac{p}{l}}$$

As, moreover, we saw in the first half of this note that η_m is dependent only upon p, R and l as a function of

$$\frac{p.R}{2.l}$$

the following may also be written for this :—

$$\frac{p.R}{2.l}\,\frac{1}{4\eta_m}\,\frac{\delta\eta_m}{\delta\frac{pR.}{2.l}}\text{ or }F_R\,\frac{1}{4\eta_m}\,\frac{d\eta_m}{dF_R}$$

so that

$$\frac{1}{\eta_R}=\frac{1}{\eta_m}\left(1-\frac{F_R}{4\eta_m}\frac{d\eta_m}{dF_R}\right)=\frac{1}{\eta_m}\left(1-\frac{d\ln\eta_m}{4d\ln F_R}\right)$$

This formula can, therefore, at once be applied to the η_m–F_R curve, irrespective of the particular values of R. p and l, thus to a series of observations made with one capillary under various pressures.

It will, however, always be desirable to make series of determinations with at least two capillaries, for the detection of the presence of disturbing influences.

Note 2.—To this the following can be added :—

(1) The viscosity was calculated with the following formula (see, for instance, Hatschek) :—

$$\eta = \frac{P_g D}{4\pi L} \frac{1}{\Omega} \left(\frac{1}{R_1^2} - \frac{1}{R_2^2} \right) 0{\cdot}92$$

in which

L=Height of the asphaltic bitumen in cylindrical part.

Ω=Angular velocity in radians per second.

0·92=A correction factor for the friction in the pulleys.

For the meaning of the other symbols see Fig. 7.

(2) The influence of the bottom, irregularly formed part was eliminated by making determinations with two different levels of filling, *e.g.*, to *a* and to *b*, for if the weights P_a and P_b are necessary to get the same Ω, $P=P_b-P_a$ is then necessary for the rotation of part a—b, the difference in height of which is L.

(3) It was particularly when there was a great difference between R_2 and R_1 that the surface of the asphaltic bitumen did not always remain straight at great turning speeds, but slanted (c. Fig. 7). For this reason, therefore, operations were carried out with small R_2-R_1, viz., $R_2=3{\cdot}16$ cm. and $R_1=2{\cdot}50$ cm. (The cause of the phenomenon here described is unknown to us.)

(4) On inside and outside cylinders the same moment works per 1 cm. height; the surfaces are, however, different, so that here, too, the shearing stress is not everywhere the same. The maximum and minimum values are in the ratio of $R_2 : R_1$, hence, with the above dimensions, 1·26 : 1. We have assumed that the shearing stress corresponding to the arithmetical mean radius belongs to the (average) viscosity obtained.

Note 3.—This formula can be approximately deduced with comparative simplicity :—

If at the moment t we call the penetration depth l, then the resistance experienced by the penetrometer needle is the sum of two parts :—

1. A resistance at the point of the needle. This we accept as constant (w_1).

2. A resistance at the side of the needle, which we suppose to be proportionate to l ($w_2 l$).

Total $w_1+w_2 l$. The rate of penetration is, therefore :—

$$\frac{dl}{dt} = \text{constant} \times \frac{P}{\eta} \times \frac{1}{w_1+w_2 l} = \frac{P}{\eta} \times \frac{1}{a+bl},$$

in which P represents the load of the needle. Integration (with t=o let us take l=o) yields :—

$$(1)\quad al + \tfrac{1}{2}bl^2 = \frac{Pt.}{\eta}$$

With P=100 grams and t=5″, l is the normal penetration. Experimentally we found :—

$$(2)\quad \text{pen.}^{1.94} = \frac{\text{constant.}}{\eta}$$

As, particularly with larger penetrations, the term a. can only be a correction term, (1) and (2) tally well.

Further, from this deduction there follows a relation between penetration and P and t : so long as Pt is constant the penetration must remain the same. The following table XII.—recording determinations with a non-plastic asphaltic bitumen at the same temperature—does not altogether bear this out, for with P equalling 200 grams or more, deviations occur. Consequently, determinations with such high loads cannot be simply converted ; complications apparently occur.

TABLE XII.

Relation between Penetration and Duration and Load.

P in grammes.	t in seconds.	$\frac{Pt.}{50}$	Penetration.	$\frac{pen.}{\sqrt{\frac{P}{50}t.}}$
50	1	1	11	11
100	1	2	16	11
200	1	4	27[5]	14
50	2	2	16	11
100	2	4	21	11
200	2	8	36[5]	13
50	5	5	24[5]	11
100	5	10	30	10
200	5	20	57	13
400	5	40	87	14
50	10	10	32	10
100	10	20	49	11
200	10	40	83	13
50	20	20	50	11
100	20	40	68	11
200	20	80	110	12

The above deduction again applies solely to non-plastic asphaltic bitumens. For plastic bitumens see Hubbard.[4]

Note 4.—The determinations recorded in the table were carried out with an inside cylinder radius of 2·50 cm. and an outside cylinder radius of 3·16 cm. A series of determinations was also

[4] *J. Agri. Res.*, 5, 805–818.

made with an inside cylinder radius of 2·00 cm. Complete agreement was found between the two series. For the possibility had existed that the asphaltic bitumen would not adhere sufficiently to the side of the apparatus, with the result that the bitumen might have slipped along the wall. The agreement between the results with the two apparatus, however, excludes this possibility.

The absence of slip also in the case of the other asphaltic bitumens was proved in this way.

Note 5.—An explanation may be found in various directions :—

(1) *Influence of the Reservoir.*—The apparatus was calibrated with a liquid of constant viscosity. On account of the low rates of shear, the viscosity is high and consequently gives the reservoir relatively more resistance. A test, however, showed that the reservoir can at the most absorb 5 per cent. of the applied pressure with the widest capillary ; so it is no use seeking for an explanation in influence of the reservoir.

(2) *Slip*, through the asphaltic bitumen's gliding along the walls.

This is unlikely for the following reasons :—

(*a*) The shearing stress is comparatively slight ; the highest applied figure is about 400 gr. per sq. cm. In most cases this force is not above 100 gr. per sq. cm.

(*b*) In determinations with the Couette viscometer (shearing stress up to 10 gr. per sq. cm.) the absence of slip was proved.

(*c*) Glass and metal capillaries of the same dimensions yielded the same viscosity figure. If there were any slip, this would probably also be influenced by the solid wall.

(*d*) It follows from the complete agreement between asphaltic bitumens A and B with the two apparatus that slip does not occur there.

Moreover, it is apparent from the following calculation that the position of the curves in Fig. 11 can certainly not be explained only by slip.

The slip coefficient with different shearing stresses at the side need not be the same ; the two capillaries must therefore be compared with equal $\frac{p.R.}{2.l}$

Then, at the same time, the η_m values must be the same. A volume as follows then flows through a capillary per second :—

$$V = \frac{\pi.p.R^4}{8.\eta_m.l} + \lambda_{\frac{p.R}{2.l}} \pi R^2 . \frac{p.R}{2.l} = \frac{\pi.p.R^4}{8.\eta'.l}$$

in which

η_m = the actual (average) viscosity.

$\lambda_{\frac{p.R}{2.1}}$ = the slip coefficient for a shearing stress $\frac{p.R.}{2.1}$

η' = the measured (average) viscosity.

By simplification we get :—

$$\frac{1}{\eta'} = \frac{1}{\eta_m} + \frac{4\lambda F}{R}$$

so that the measured viscosity must steadily decrease with the radius of the capillary, compared at equal $\frac{p.R.}{2.1}$ This is not the case in Fig. 11, so that in any event there are other influences.

Summarising, therefore, we come to the conclusion that the absence of slip cannot be definitely proved, but can be shown to be probable.

(3) There remains elasticity or thixotropy.

We were able to indicate the presence of thixotropy definitely by observations with capillaries of varying length. These are recorded in the following table, in which p is expressed in kilos per sq. cm., and η_m in k.p.

R = 0·107 cm.						R = 0·242 cm.					
l = 4·9 cm.			l = 25 cm.			l = 4·9 cm.			l = 25 cm.		
p	η_m	$\frac{p}{\eta_m}$	p	η_m	$\frac{p}{5{\cdot}1\eta_m}$	p	η_m	$\frac{p}{\eta_m}$	p	η_m	$\frac{p}{5.1\eta_m}$
5	37·8	0·132	15	38·5	0·076	5	11·0	0·455	15	15·2	0·193
7·5	24·6	0·305	25	20·8	0·235	7·5	7·8	0·96	25	7·9	0·62
10	17·6	0·57	35	12·9	0·53	10	5·3	1·89	35	5·05	1·36
			50	8·15	1·21				50	3·1	3·1

To this must be added :—

(*a*) The viscosities were corrected for the influence of the pressure by means of the observations recorded later in the paper.

(*b*) Asphaltic bitumen A, determined with these long capillaries and also corrected for pressure influence, tallied with the earlier figures ; so evidently the great energy applied does not cause increase in temperature.

(*c*) $\frac{p}{\eta_m}$ and $\frac{p}{5{\cdot}1\eta_m}$ indicate the quantities of liquid flowing through per second in an arbitrarily chosen measure.

For from Poiseuille's formula it follows that

$$V=\frac{\pi R^4}{8}\cdot\frac{p}{\eta_m l}, \text{ so that } \frac{p}{\eta_m} \text{ and } \frac{p}{\eta_m\cdot\frac{25}{4\cdot9}}=\frac{p}{5\cdot1\ \eta_m}$$

are proportionate to the V's.

If these volumes are plotted against $\frac{p}{l}$, the slope in pressure in the capillary, the following are found to be necessary for the efflux of equal volumes :—

R = 0·107 cm.		R = 0.242 cm.	
l = 4·9 cm.	l = 25 cm.	l = 4·9 cm.	l = 25 cm.
p = 5.0 kgs/cm²	p = 5·1 × 3·7	p = 5·0 kos/cm²	p = 5·1 × 4·25
7·5	5·1 × 5·5	7·5	5·1 × 6·1
10·0	5·1 × 7·2	10·0	5·1 × 8·05

In the first set of capillaries the relation of the pressure differences is 0·74 ; 0·73 ; 0·72; and in the second : 0·85 ; 0·82 ; 0·81.

The only possible explanation is that the viscosity diminishes as a result of flow, in other words, that the asphaltic bitumen is thixotropic (for elasticity would produce a converse effect).

In the observations of Fig. 11, elasticity also possibly plays a part.

It should here be pointed out that thixotropy cannot be proved with certainty to be absent by means of a graph as in Fig. 11, for as, with equal $\frac{p.R}{l}$ the η_m value first decreases and then increases as R increases, it is possible that in two series of observations these by chance coincide. A certain method is, however, provided by two capillaries of different length.

It is difficult to indicate the degree of thixotropy. A viscosity determination requires flow of the liquid for some time, during which time an average of the (decreasing) viscosity of the thixotropic liquid is determined. For instance, if in the case of two asphaltic bitumens, I. and II., the course of the viscosity during the period of the determination is similar to that represented in Fig. 12, it will depend upon the duration of the determination as to which of the two seems to be the more thixotropic. If this duration is t_1, then in the case of I. the proportion of the determined viscosity η_1 with respect to the final value η_2 will be greater than that between η_3 and η_4 in the case of II. If, on the other hand, the duration is t_2, then the reverse is possible.

If, therefore, in future, we say that an asphaltic bitumen I. is more thixotropic than an asphaltic bitumen II., this should really be taken to mean that it is more thixotropic for the viscometer we have used.

Note 6.—Some properties of the bitumens used are given in Table XIII.

TABLE XIII.

	A.	B.	C.	D.	E.	F.	G.	H.	I.
Specific gravity 25/25° C.	1·01	1·02	1·02	1·03	1·01	1·01	1·03	1·04	1·06
Melting point R. & B. °C.	33	50	39	85	43	39	41½	57½	47
Pen. 100 gm/5" at 0° C.	5	—	15	9	28	22	18½	6	9
,, ,, 15° C.	71	4·5	65	19	98	95	67	20	4
,, ,, 25° C.	>250	28·5	178	34	190	242	198	48	26½
,, ,, 40° C.	—	>250	>250	61	>250	>250	>250	164	190
,, ,, 50° C.	—	—	—	108	—	—	—	>250	>250
,, ,, 60° C.	—	—	—	180	—	—	—	—	—
,, ,, 75° C.	—	—	—	>250	—	—	—	—	—
Ductility at 0° C. ..	0	0	35	0	6	36·5	50	0	0
,, 15° C. ..	>100	>100	>100	2·5	44	>100	>100	58	0
,, 25° C. ..	—	—	—	3	82	—	—	>100	>100
,, 40° C. ..	—	—	—	5	>100	—	—	—	—
,, 50° C. ..	—	—	—	6	—	—	—	—	—
Solubility in petroleum spirit 60/80° C. %	99	99	87	68	79	83	80	76	88

Combustion and Injection in High-Speed Compression-Ignition Engines.*

By D. A. C. Dewdney (Student).

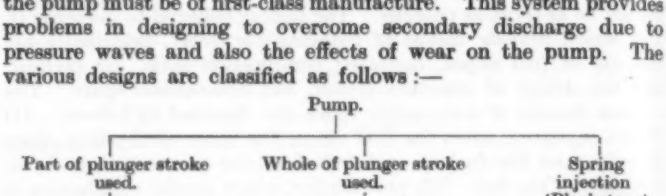
The paper deals first with combustion and then with injection. The process of combustion in the compression-ignition engine has been described by Ricardo, who divides it into three parts: (1) The delay period, before the fuel starts to burn; (2) the period of rapid pressure rise; (3) the period of controlled burning. Smooth running is dependent largely upon the length of period (1), which should be as short as possible. The factors believed to influence it, and which provide means of reducing the delay, principally are the use of fuel dopes, increased compression ratio, air preheat, and the design of injection system and combustion space. The various designs of combustion space are classified as follows: (1) Direct injection, when the fuel enters the main combustion space directly, and the fuel spray is relied on for penetration and distribution of the fuel; (2) prechamber, where partial combustion of the fuel in an auxiliary chamber provides the fuel velocity necessary for complete distribution; (3) air chamber type, where air is compressed into a separate chamber on the compression stroke, and during the expansion stroke is released into the combustion chamber to assist combustion. These three classes are further sub-divided according to the type of induced turbulence—namely: (*a*) port swirl; (*b*) piston swirl, and (*c*) divided chamber type. Type (1) provides easy starting, but high injection pressures are necessary and the injection timing requires alteration with speed, which itself is limited by considerations of the rate of propagation of fuel. When sufficient turbulence is induced, these disadvantages are lessened. Type (2) suffers from high heat loss, so that high combustion pressures are necessary, tending to a heavy engine. Type (3) has a smaller power output per volume, owing to the fact that the explosion power is developed relatively late in the stroke.

Curves have been plotted showing the effect of compression ratio, volumetric efficiency and combustion efficiency on combustion performance. The M.E.P. is increased by increase in compression ratio, volumetric efficiency and efficiency of combustion. Increase in compression ratio is followed by increased losses, and a practical gain is hard to realise. Increase in volumetric efficiency is only advantageous if the fuel distribution remains good, when the increased air charge results in more efficient combustion. A large air excess reduces efficiency.

The process of ignition is not yet well explained. Callendar's nuclear theory, in which hydrocarbon peroxides are considered

* Précis of paper submitted for Students' Medal and Prize, 1931–1932.

to be intermediate products of combustion, offers the most generally satisfactory explanation. The injection system in a compression-ignition engine is most important, and accuracy in metering and timing control the efficiency and smooth running of the engine. Injection systems are classified as : (1) Storage system ; (2) timed pump. In the former the fuel is raised to the injection pressure by a pump, and the timing and metering is done by the injection valve ; in the latter, compression, timing and metering are all done by the pump. The latter method is more widely used, but the pump must be of first-class manufacture. This system provides problems in designing to overcome secondary discharge due to pressure waves and also the effects of wear on the pump. The various designs are classified as follows :—



The injection nozzle must be considered in conjunction with combustion chamber and pump. The effects of charges of design on the type of spray have been investigated. The nozzle controls the shape of the fuel spray, the penetration and the degree of atomization. The two latter factors are incompatible, and compromise is necessary. The spray angle from a parallel-sided jet is not more than 15°, and 12° is the maximum in practice. The angle can be increased by using a centrifugal spray, and better distribution in direct engines can be obtained in this way without the use of a number of small holes in the flame plate. The formation of the spray is also considered. It is believed that the spray is formed by disintegration of the jet of oil owing to the difference in velocity of fuel and air, and Lord Rayleigh's work on the collapse of liquid columns is applied in support of this theory.

Messrs. Farmer and Alcock have analysed mathematically the flow of fuel in the injection system, and curves can be drawn showing the pressure rise in the system for various types of injection pumps and nozzles. The question of secondary reflected waves in the fuel pipes and their effects on engine performance is also mentioned.

The Occurrence, Nature and Origin of Asphaltites in Limestone and Oil Shale Deposits in Estonia.

By Paul N. Kogerman, M.Sc., D.I.C. (Member).

Introductory.

The occurrence in limestone beds of Estonia of dark-coloured, non-volatile species of solid bitumen, mostly in the form of veins and lenses, from 7 to 9 in. in diameter and from 2 to 3 in. in thickness, was reported by a geologist nearly a century ago.[1]

Prof. C. Grewingk, in his review of mineral resources of Baltic States, records an occurrence of small lumps of *anthracite* in ordovician *vaginatum* limestone at Narva, and of *asphalt* in silurian limestone on the Isle of Dagö, in ordovician greenclay at Paldiski (Baltic Port) and in some other places. No analytical data on the composition of these minerals were given.[2]

H. v. Winkler[3] and B. Doss[4] assumed that the so-called "anthracite" might be an asphaltite.

On the basis of a chemical analysis of the asphalt-like bitumen, Doss assumed it to belong to *grahamites* (*Cl. Richardson's* classification), but it differs from typical grahamites by its brown colour in pulverised form and its high solubility in turpentine oil (about 70 per cent.).

An analysis of a specimen of this black, lustrous, brittle mineral was as follows[4]:—

Sp .gravity 1·11–1·17 at 18·2°C.

Ultimate composition:—

C	83·28	per cent.
H	8·53	„ „
S	2·16	„ „
N	1·51	„ „
$O+H_2O$	3·81	„ „
Ash	0·71	„ „

[1] *v. Eichwald*, E. Urwelt Russlands, H. II., S. 17, 1842—*Schrenk*, A.G., Übersicht des obersilurischen Schichtensystems Liv- und Esthlands usw. T.I. Arch. f.d. Naturkunde Liv-, Esth- und Kurlands, Ser. I, 1 16, 37 Dorpat 1854. *Oserky*, G., Georgr. Umrias des nordwestlichen Esthlands, Verh. d Kais. Min. Gesell. zu St. Petersburg, 1844, p. 26.

[2] *Grewingk*, C., Übersicht der Mineralien und Gesteine Liv-, Est- und Kurlands und ihre Nutzbarkeit, *Sitzungsberichte d. Naturforscher-Gesellschaft*, Dorpat, 1866, pp. 52-53.

[3] *v. Winkler*, H., Über Umfang und Abbauwürdigkeit estländischer Bodenschätze. Mitt. a. d. Geol. Inst. d. Univers. Greifswald, 1920, s. 10.

[4] *Doss, B.*, Ein Vorkommen von Grahamit im Silurkalk von Kunda, *C.f. Min.*, 1914.

Solubility in common organic solvents :—

Ethyl alcohol (96 per cent.) ..	1·5	per cent.
Acetone	10·0	,, ,,
Ether	29·0	,, ,,
Terpentine oil	71·0	,, ,,
Carbon disulphide	92·0	,, ,,
Chloroform	97·0	,, ,,
Carbon tetrachloride	51·0	,, ,,

Another specimen of an asphalt-like bitumen from Estonian blueclays at Kunda (Lower Cambrian) was analysed by Karpinsky.[5] The analysis was as follows :—

C	77·23	per cent.
H	8·95	,, ,,
N	1·10	,, ,,
O	7·52	,, ,,
Ash	5·20	,, ,,
Sp. gravity at 19·3°	1·118	,, ,,

According to Karpinsky, the mineral resembles an *albertite*. Finally, Cunningham Craig[6] mentions an occurrence of "thin vertical veins of manjak" in limestone quarries near Vanamoisa. Of all these findings reported so far, and recently reviewed by Dr. L. Rüger,[7] but few have been analysed. The chemical nature of these bitumens, and even their place amongst other minerals, has not been determined with any degree of certainty by the previous scientists. The same uncertainty is observed in all attempts to explain the origin of the bitumens. Scupin[8] did not know of the occurrence of asphaltites in kukersite (Estonian oil shale) beds, and assumed that the bitumen might have been derived from bituminous *Dictyonema* shale. Cunningham Craig applies his inspissation theory to explain the formation of the bitumen even in this case. He says[9]: "Possibly the intrusion of these manjak veins took place at a later period than the impregnation of the shale."

A study of the literature on analogous findings in Scandinavian countries reveals the fact that lenses of solid bitumen or "coals,"

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[5] Rüger, L., Baltische Staaten Erdöl-, Asphalt-und Erdgas-vorkommen, Engler-Höfler, Das Erdöl, II., 2 Teil, 2 Aufl., pp. 420-422.

[6] *Cunningham Craig, E. H.*, Kukkersite, "The Oil Shale of Estonia," *J. Inst. Petr. Techn.*, 1922, **8**, 357-8.

[7] *Rüger, L.*, loc, cit.

[8] *Gäbert, C.*, and *Scupin H.*, Bodenschätze im Ostbaltikum (Die Kriegsschauplätze, 1914-1918, geol. dargest. H.10, T. II., S. 75) Berlin, 1928.

[9] Loc. cit.

as the bitumen is called, have been found in Cambro-Silurian deposits at Västergötland (Sweden) as well.[10]

The chemical composition of these bitumens varies between the following limits :—

C	.. ..	73—87 per cent.
H	.. ..	4—5 ,, ,,
N+S	.. .. .. about	2 ,, ,,

A specimen of so-called " Ödergarden coal " from the *Petlura* horizon had the following ultimate composition :—

C	82·68 per cent.
H	5·02 ,, ,,
N	1·84 ,, ,,
S	2·92 ,, ,,
O (by difference)	2·09 ,, ,,
Ash	5·45 ,, ,,

Vegetable débris have not been found in the Swedish bitumen, and Schreiter, following the hypothesis of Baragwanath (an explanation of the origin of veins of asphaltites at Minas ragra in Peru) assumes that the coals of Västergötland might have been formed on metamorphosis of petroleum. The author (Schreiter) draws attention to the high vanadium contents of the mineral, and suggests a connection between the radioactivity of vanadium compounds and the formation of the solid bitumen.

Part I.

Properties of Estonian Asphaltites.

The development of oil shale mining in Estonia revealed the fact that veins and lenses of asphaltites occur in the oil shale deposits as well, and that their occurrence is not uncommon. The present author had the privilege to investigate the first specimens of solid bitumen from M.-ordovician oil shale beds found at Kukruse and Kävа (Kohtla) mines.[11]

Further, the author re-analysed the specimens of " coals," obtained from the collections of the Geological Museum at this University and mentioned in C. Grewingk's review.[12]

The biggest lump of asphaltite analysed (Figs. 1 and 2) was about 25 cm. in length and 2-5 cm. in thickness. It formed probably a lense in the shale bed. The shale layers are almost horizontal, very regular in stratification without any signs of faults or other deformation. The change from yellowish-brown shale to black lustrous asphaltite is usually sharp and distinctive,

[10] *Schreiter, R.*, Kambro-Silurische " Kohlen " von Västergötland, Sweden, Z.d.Deutsch. Geol. Gesellschaft, 1931, 88, H. 9, 635-641.

[11] See the Section of Kohtla mine : P. N. Kogerman, " The Present Status of Oil Shale Industry in Estonia," *J. Inst. Petr. Techn.*, 1925, **11**, 213.

[12] Loc. cit.

but in some spots the change is more gradual (Fig. 3). In the mass of the asphaltite one can find here and there fragments of the same shells that occur in the shale.

The composition of the layer of air dry oil shale in which the bitumen is embedded, and those between which it lies, is given in the Table I.:—

TABLE I.

Composition of the Layers of Oil Shale in which the Asphaltite Occurs.

	Per cent.				
	Total moisture.	Hygroscopic moisture.	Ash.	CO_2.	Organic matter.
A.—Average sample of the layer in which the asphaltite is embedded ..	2·05	0·04	24·84	4·70	70·46
A^1.—The same layer picked pieces without grains of limestone	5·44	0·04	21·48	1·52	77·00
B.—Average sample of the bottom and top layers ..	3·46	0·14	39·08	12·50	48·42

On distillation (Fischer's method) the sample A^1 yielded: 3·5 per cent. of water, 54·8 per cent. of oil, and 30·3 per cent. of coke and ash.

The physical properties and the composition of the bitumen were as follows[13]:—

1. Colour in mass	black
2. Fracture	hackly
3. Lustre	bright
4. Streak on porcelain	brownish-black
5. Specific gravity at 15° C.	1·11
6. Hardness, needle penetrometer at 25° C.	0
7. Fusing point (K. and S. method)	(above) 330° C.
8. Fixed carbon	37·22 per cent.
9. Soluble in carbon disulphide	93·50 ,, ,,
10. Soluble in benzene	86·87 ,, ,,
11. Mineral matter	3·86 ,, ,,
12. Non-mineral matter insoluble in carbon disulphide	2·64 ,, ,,
13. Solid paraffins	none
14. Diazo-reaction	negative
15. Acid value	0·2
16. Ultimate composition :—	
C	84·85 per cent.
H	8·62 ,, ,,
S	0·53 ,, ,,
O+N (diff.)	2·14 ,, ,,
Ash	3·86 ,, ,,

[13] The tests referred to here are described in H. Abraham's book, "Asphalts and Allied Substances," New York, 1929.

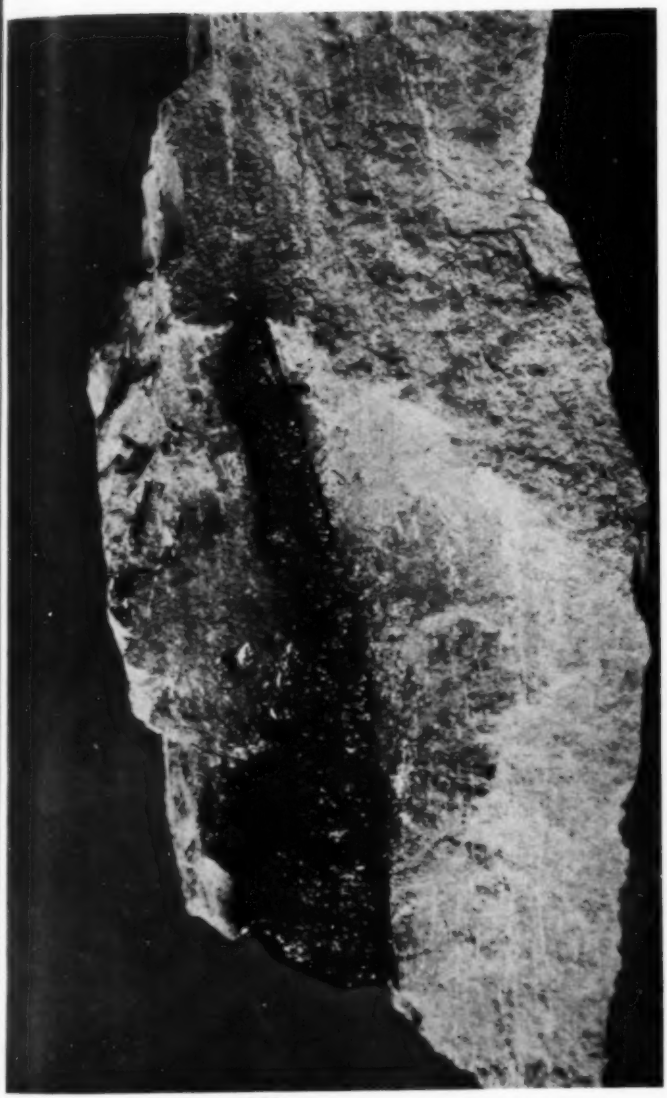

FIG. 1.

A LENSE OF ASPHALTITE IN KUKERSITE SEAMS. FRONT VIEW—WHITE SPOTS IN ASPHALTITE ARE CAUSED BY REFLEXION OF LIGHT.

FIG. 2.
THE SAME LENSE (AS FIG. 1). BACK VIEW.

FIG. 3.

A THIN VEIN OF ASPHALTITE IN BITUMINOUS LIMESTONE.

FIG. 2.

THE SAME LENSE (AS FIG. 1) — BACK VIEW.

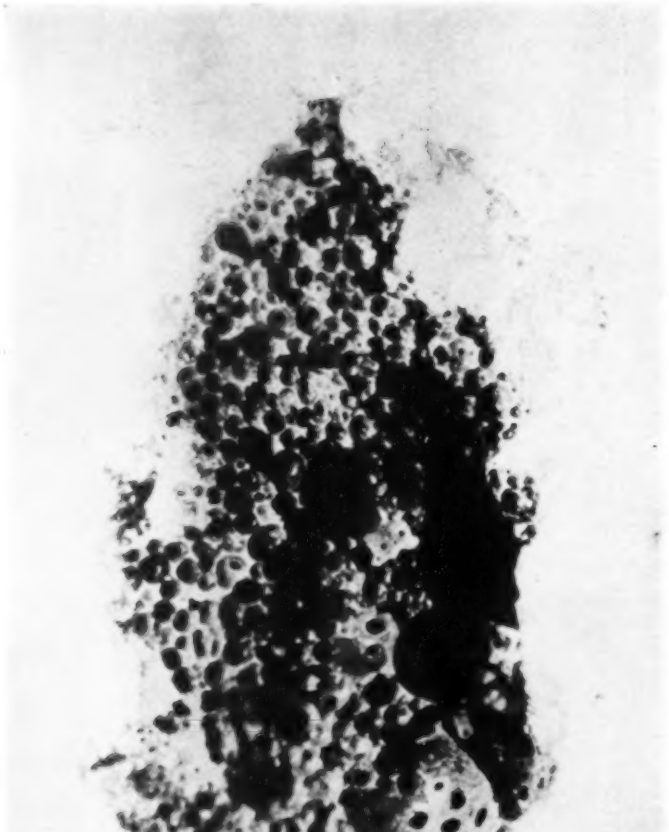

FIG. 4.

SURFACE OF AN ASPHALTITE FROM LYCKHOLM LIMESTONE (MAGNIFIED 20 DIAMETERS).

The mineral matter does not contain any vanadium-compounds, nor does it show radioactivity.

On "Fischer-distillation" the bitumen frothed, drops of water and of a very viscous substance, which on cooling solidified and became brittle and pitch-like, passed over.

On heating *in vacuo*, under 6 mm. Hg, the bitumen was foaming and slowly decomposing at about 335° C. without yielding any oil.

Another specimen of the bitumen from Käva (Kohtla) mine was analysed by Mr. K. Luts at Kohtla Laboratory. The mineral was extracted with carbon disulphide, and the extract had the following composition :—

C	87·90 per cent.
H	8·89 ,, ,,
O+S (by diff.)	3·21 ,, ,,
Iodine value	34

Drops of black bitumen, which occur in the cavities of fossils (*Orthoceras*), found in *Megalaspis* limestone at Narva, and described by older authors as "tears" or "bright coal," had the following ultimate composition :—

	Average sample. Per cent.	Ash-Free. Per cent.
C	81·04 ..	84·78
H	8·27 ..	8·66
O+Sen. (by diff.)	6·27 ..	6·56
Ash	4·42 ..	—

The bitumen is soluble in CS_2.

On the basis of the analytical data there is no doubt that the bitumen found in Estonian limestone and oil shale is an *asphaltite*. The properties of the mineral correspond closely to those of a grahamite. From correlation of geological and analytical data it seems to the author that the "coals" of Västergötland[14] might be asphaltites as well.

Part II.

Origin of Estonian Asphaltites.

1. The generally accepted hypothesis of origin of native asphalts and asphaltites[15] assumes that asphaltites have been formed from petroleum by oxidation and polymerisation. As a result of this hypothesis, native asphalts and asphaltites have been classified by C. Engler[16] as an "oxybitumen." The same view is shared also by other scientists.[17]

[14] Loco cit.

[15] (1) *Abraham, H.*, Asphalts and Allied Substances, New York, 1929. (2) *Engler, C.* und. v. *Höfer, H.*, Das Erdöl, Bd. I., 1912, p. 36 (3) Marcusson, J., Die natürlichen und künstlichen Asphalte, 1931, p. 15.

[16] *Engler, C.*, and v. *Höfer, H.*, loco cit.

[17] *Hackford, J. E.*, "The Significance of the Interpretation of the Chemical Analysis of the Seepages," *J. Inst. Petr. Techn.*, 1922, **8**, 200-201.

Fig. 4.
SURFACE OF AN ASPHALTITE FROM ENCRINITE LIMESTONE (MAGNIFIED 20 DIAMETERS).

In the case of Estonian asphaltites the possibility of intrusion of petroleum into the compact white limestone and into the shale is out of the question. The geological investigations do not support the hypothesis of an impregnation of limestone or shale deposits. Assuming that petroleum had impregnated an old Ordovician shale strata producing a rich oil shale, which in extreme cases contains only about 25 per cent. of mineral matter, evenly distributed in the organic substance, then one must imagine a very porous calcareous seam, but strong enough to withstand the pressure of the overlaying hard limestone strata, prior to the impregnation. This assumption could hardly be supported by geological evidence. On the other hand, the occurrence of pieces of asphaltite "incapsulated" in cavities of fossils in impervious compact limestone, is such a strong evidence against intrusion of petroleum that we must search for other explanations of origin of asphaltites in this case.

The only plausible explanation of the genesis of Estonian asphaltites is, that the mineral has been formed *in situ* directly from the original organic substance of bottom sediments. The organic sediments are the source of the oil shale as well.[18]

On certain spots, under special conditions of decomposition, the protobitumen, as the original organic matter of sediments is called by R. Potonié,[19] had been undergoing a change in two directions: *i.e.*, one leading to the oil shale, the other to the asphaltite:—

Protobitumen → Oil shale
Protobitumen → Asphaltite

In the case of limestone deposits, the organic matter of animals (*Orthoceras* and others) had been undergoing the same transformation.

The possibility of formation of an asphaltite from a pre-formed oil shale is doubtful because of the different ash contents of the two minerals (see Table I.), and the absence of signs of metamorphosis of the oil shale.

2. Comparing the ultimate compositions of organic sediments, kerogen of kukersite, blown-asphalts from shale oil, and asphaltite, one cannot fail to observe a close similarity in the composition of Estonian asphaltites and asphalts from kukersite oil (see Table II.). In the series—protobitumen, kerogen, shale oil, asphaltite—the

[18] For particulars, see *Kogerman, P. N.*, "The Chemical Nature of Estonian Oil Shale (The Origin of Oil-Shales)," Sitzungsberichte d. Naturforscher-Gesell. zu Tartu, 1927, XXXIV, 2.

[19] *Potonié, R.*, Allgemeine Petrographie der "Ölschiefer" und ihrer Verwandten, Berlin, 1928.

carbon-hydrogen ratio gradually increases. In the case of oil shale the transformation of protobitumen seems to have been stopped at an intermediate stage. On the basis of this assumption the term "oxybitumen," in the sense of Engler, cannot be applied to the asphaltite.

What factors have been acting in the process of formation of Estonian asphaltites? As already mentioned above, the radioactive substances are absent. Considering the limnological and limnogeological facts, one naturally must look for biochemical agents. A study of the microphotographs (Fig. 4) of the surface of an asphaltite from limestone leaves but little doubt that the organic sediment (or organic matter of bottom dwellers) had been rapidly decomposing under the action of bacteria with strong evolution of gases.[20] The small pieces of asphaltite have a distinctly "foamy" or "bubble" structure.[21]

In its early stage the whole system of degradation products formed probably a huge "bubble," covered with a thin skin of organic decomposition products; the pressure of the gases inside had prevented the intrusion of mineral matter into the lense.

There might have been variations in the composition of the organic sediments as well.

TABLE II.

Comparative Percentage Composition of Organic Sediments, Kerogen of Kukersite, Asphalt from Shale Oil and Asphaltites.

	Mud of Mid-Russian lakes.[1]	Coorongite.[2]	Kerogen of Kukersite.[3]	Vacuum Oil from Kukersite.[3]	Refined Bermudez Asphalt.[4]	CS_2 Extract of Asphaltite (Estonian).	Blown-asphalt from Kukersite Oil.[3]	Estonian Asphaltite.
C ..	50–60	64·7	76·6	83·3	82·9	87·9	83·9	84·8
H ..	7–9	11·6	9·1	9·7	10·8	8·9	8·7	8·6
O+N+S	30–35	23·7	14·3	7·0	6·3	3·2	7·4	6·6
C/H ..	5·5–7	5·6	8·4	8·6	8·0	9·9	9·6	9·9

Bibliography.

[1] Dexbach, H.K., "The Bottom of Cossino Lakes (in Russian), Moscow, 1925, 8.
[2] Twenhofel, W. H., "Treatise on Sedimentation," Baltimore, 1926, 301.
[3] Kogerman, P. N., "On the Chemistry of the Estonian Oil Shale 'Kukersite,'" Tartu, 1931.
[4] Abraham, H., "Asphalts and Allied Substances," New York, 1929, 102.

However the difficulties of the explanation of transformation reactions here are of the same order as in the case of theories of origin of anthracite and its relation to the bituminous coal.

In one case the decomposition of protobitumen had been stopped at the kerogen stage, in the other it went right through to the asphaltite stage. On mild heat treatment of the kerogen or by air-

[20] These decompositions have probably caused the accumulation of natural gas, the evolution of which has been observed at the north coast of Estonia, in the area in which the asphaltites occur.

[21] It is worthy of notice that the microphotographs of thin horizontal sections of kukersite (X390) show a similar "vesicular-structure." See R. Potonié's book, Fig. 16.

blowing of the primary shale oil a solid bitumen similar to the asphaltite can be produced. The author has not the intention to generalise his conclusions, but simply emphasises that the problem of genesis of asphalts and asphaltites needs further study before the veil that prevents seeing the *nature's ways* of oil and asphaltite production could be disclosed.

Conclusions.

1. A solid bitumen from Estonian limestone has been analysed and its properties determined.

2. Specimens of solid bitumen found in Estonian oil shale have been analysed; their resemblance with the bitumen from limestone has been established. These species of bitumen are asphaltites.

3. It is assumed that the asphaltites have been formed *in situ* from organic sediments by biochemical changes in parallel (synchronically) to the oil shale formation.

The author desires to record his thanks to Prof. A. Öpik for photos. No. 3 and 4, and to Mr. E. Kilkson for determining of the radioactivity of the minerals.

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Discussion on "Some Observations on the Study of an Oilfield,"* by J. W. Weil.

The following comments, perforce submitted in writing, are offered as a contribution to the points discussed in Mr. Weil's paper, and subsequently in the general discussion. Criticism of so valuable a paper must be limited to that of Mr. Dewhurst as to modest length with which the author contented himself.

The old controversy as to palæontological versus other methods of correlation peeped out during the discussion, and is also referred to by the author in the paper. Surely, in regard to these matters, the correct view point is to keep an open mind, and to be prepared to apply *any* method or scientific aid that may best suit the local conditions. Thus, if we take the two main branches of microscopical work that are called in to aid the oilfield geologist—micro-palæontology and the study of the "heavy" minerals of the sediments—each has its own sphere in which from preliminary reasoning it is likely to be more accurate than the other, and practical experience bears this out. Palæontology is likely to be the more satisfactory in deeper water beds, and beds of some geological age, where the mineral assemblage is likely to be fairly uniform through a considerable thickness of beds. On the other hand, in the soft estuarine and near-shore sediments of Tertiary age, which play so important a part in many of the world's larger oilfields, the mineral assemblages usually show a fairly rapid vertical variation, and within a small area, such as an oilfield, particular mineral zones are more likely to represent true stratigraphic planes, since they depend on erosion and deposition within a short period, than, say, Foraminifera, which frequently tend to be facies fossils.

The writer is under the same disability in writing these comments as handicapped the author in the preparation of the paper, but the following instance can fairly be given as illustrating the point made above, and also the value of the Schlumberger method. The field concerned was in soft Tertiary beds, and the writer and his colleagues had made a careful study of the "heavy mineral" assemblages, ascertained markers for correlation, and from these had mapped faulting in the field. This correlation was at variance

* *J. Inst. Petr. Techn.*, 1932, XVIII, 881–897.

with that given by palæontology. Mr. Weil subsequently correlated the field from lithological and field considerations only, and agreed almost precisely as to stratigraphy and structure with the results obtained by the petrological work. Schlumberger diagrams of certain wells became available after both correlations had been completed, and were found to agree precisely, both in intervals between markers (*i.e.*, in stratigraphy) and in structure and the display of faults, with the work of Mr. Weil and the writer.

On the other hand, in an older Tertiary limestone-sand field, the writer found micro-petrology of no value, but that micro-Foraminifera provided adequate horizons for the correlation of an otherwise difficult field.

With regard to Mr. Garrett's criticism of the author's suggestion that it is desirable to know the changes in apparent thickness, etc., in the upper barren portion of a well, of which our knowledge in mechanically cored wells is practically *nil*, surely it is clear that with such knowledge at our disposal we can ascertain the presence, position, and hade of faults before they reach sufficient depth to affect the producing sands. We are thus saved expensive failures at new locations, and may also be saved from mistakes in drilling in the wells in which such faults cut the higher barren strata only. The writer recalls one such case in a field in which a marker some considerable distance above the producing formation was used as an indicator of the depth at which to start a very small amount of coring for location of W.S.O. This upper marker occurred as usual in the well concerned, but, necessarily unknown to the field staff, a fault of very large throw cut the well between this point and the producing sands. Fortunately it was found out by means not normally available to the field staff that the well was actually below the producing zone when coring for W.S.O.

It is in this connection that the Schlumberger method most strikingly comes into its own, since a complete section from the surface downwards can be provided for every well in the field with the loss of only a few hours per well, thus providing that accurate correlation of the upper barren beds which the author correctly states to be so necessary for the proper location and control of the drilling of wells. Whilst on the subject of faulting, the writer would invite consideration of the extent to which much apparent minor faulting may be due to crooked holes. At a time when he

had access to a very large number of records, the writer gained a definite impression that the appearance of numerous small faults on field maps had very closely coincided with the speeding up of rotary drilling, and hence of the liability to crooked holes.

The writer shares Professor Illing's dislike of cross-sections on so large a scale as 1 : 1000. Sections must be small enough for the eye and brain to get a good grasp of them. Record sections for individual wells can be on as large a scale as may be found necessary; but the writer has always found a scale of 1 : 2,500, horizontal and vertical, to be ample for cross-sections. Minor exaggeration of important narrow features is as permissible on such sections as it is on accurate maps, but the necessity for it seldom arises, since, with the individual well record sections alongside one when drawing the cross-sections, the essential points can be included, and those of less value omitted on the smaller scale.

Mr. Weil has emphasised the important point that *all* available data with reference to a well should be recorded. It is to be hoped that those training the field geologists of the future are sufficiently impressing on their students the all-important fact that their first duty is to record every, and often irrelevant-seeming, fact in connection with a well. Such facts are obtained at great expense, and are only of value if recorded at once and accurately. Deduction from facts is a fascinating exercise, and is part of one's duties on an oilfield; but it should never be forgotten that such deductions are the work of one individual, and are often transitory, whilst the faithful recording of all possible facts enables deductions to be made by a large number of workers, and may years afterwards provide the essential data for the solution of some important problem. As a slight example of this, the writer traced some diatomaceous horizons through an important key well in Egypt which had been drilled some thirty years before, when the application of the microscope to oilfield work was not dreamed of, but the samples from which had been carefully preserved by someone who appreciated his duty.

Mr. Weil also makes a significant statement on p. 882 as to the use to which all the highly expensive work done on, and in connection with, an oilfield is put. In these days of specialisation most field geologists faithfully record an enormous mass of invaluable facts and information with respect to the wells in their charge. In the very nature of things it is impossible for the field geologist himself to study all the possible facts in connection with some particular problem, since he has much routine work to do, and a time table governed by the drilling wells. If the mass of informa-

tion is sorted out by some geologist with ample time at his disposal the solution of a problem may become abundantly clear. The writer knows of a number of such cases, and would humbly offer the suggestion to oil companies of the advisability of having one or more of their geological staff with no routine duties, but with "time to think," to study problems in the light of the vast mass of data which they have accumulated at such great expense, and of which absolutely full use cannot be made on the field. The expense involved is small when considered as "overhead" on the cost of obtaining the data, whilst the financial saving effected by even the occasional solution of a problem may be very large.

T. Sutton Bowman.

Ranighāt, Nepal.

REVIEWS.

ANNUAL REPORTS OF THE PROGRESS OF APPLIED CHEMISTRY. Society of Chemical Industry, 46-47, Finsbury Square, London, E.C. 2. Pp. 721. 12s. 6d. (To Members of the Society. 7s. 6d.)

The Annual Reports of the Society of Chemical Industry on the Progress of Applied Chemistry follows the lines of the previous volumes and affords a valuable summary of the application of chemistry to industrial problems during the year 1932. The articles of particular interest to the petroleum technologist are Fuel, by Arthur Grounds (27 pages); Gas, Carbonisation, Tar and Tar Products, by J. Macleod and T. A. Wilson (31 pages); and Mineral Oils, by Alfred W. Nash and A. R. Bowen (31 pages).

LUBRICATING AND ALLIED OILS (SECOND EDITION). By E. A. Evans. London Chapman & Hall, Ltd., 1933. Pp. 175. 9s. 6d.

This book is one of the "Directly Useful Technical Series" which aims at presenting theoretical discussion with a practical bias judiciously incorporated in a practical handbook with a scientific basis.

With this object before him the author's purpose has been to explain to chemists and to engineers the meanings and implications of the terms they use when discussing lubricants and lubricant specifications. A little learning is a dangerous thing, both in a chemist and an engineer, and if it can be replaced by a little knowledge Mr. Evans will have achieved his object.

This book (which might alternatively be called "A Beginner's Guide to the Chemistry and Physics of Lubricating Oils") devotes twelve pages to the history and production of petroleum and fatty oils, one hundred and thirteen pages to a description of the physical and chemical tests usually applied to lubricating oils, eight pages to a chapter mysteriously entitled, "Oleography," which, however, does not deal with olegraphs, but with the work of Langmuir and of Hardy on boundary friction, and two final chapters to the selection of lubricants for various purposes.

On the whole, it may be said that the book fulfils its purpose quite well. Some chapters, especially the last two, are excellent and compensate for others which are "excellent in parts" or which (like the olegraphic and the historical ones) could well have been omitted.

One of the rather curious features of the book is the apparently disproportionate space which the author gives to subjects of minor importance. It is difficult to see, for example, why he should devote seven pages to describing methods of determining specific heat, and (for no apparent reason) to presenting long lists of the specific heats of individual hydrocarbons, while only half a page is allotted to artificial thickeners. The oil chemist probably has to deal with thickeners many times more frequently than he has to determine the specific heat of his oil sample. One also regrets that analytical methods which have long passed into obsolescence, or which are thoroughly unsound (*vide* p. 101), are given as much prominence as the official standard

methods which have replaced them. Very praiseworthy prominence is given to the modern procedure of recording viscosity in absolute units, but the table for inter-conversion of the different systems of viscometry is based in part on Herschell's formulæ and not the A.S.T.M. formulæ which have superseded them and in consequence the Saybolt and Engler figures are, in parts of the table, slightly in error.

The illustrations are good and excellently reproduced, but here again more careful selection would have been advantageous. Drawings of tap funnels and hydrometers are superfluous, and one doubts if Esling's Sulphur Apparatus or Gray's Flash Point Apparatus are prominent in modern lubricating oil testing laboratories.

In view of the author's great experience in lubricating oil testing and the purpose he has in view in writing the book, one regrets that he has not given one or two new chapters indicating to less-experienced chemists how they should tackle some of the common problems which they are liable to meet, such as how to examine and report on the condition of a used crankcase oil or a turbine emulsion.

A chapter on lubricant testing machines would also seem desirable, not, perhaps, because of any particular merits which have been proved for them, but because claims for their value are so frequently brought forward with greater plausibility than the ultimate results have so far confirmed. A section on cutting oils and similar "soluble oils," and a dispassionate review of the advantages and disadvantages claimed for compounded oils, would also have been valuable to those for whom this book is intended.

We are on the whole justified in hoping that the second edition of this book will command so ready a sale that a third edition, with considerable excision and revision, will soon be produced. F. B. Thole.